STIC Search Report
Biotech-Chem Library

STIC Database Tracking Number: 161714

TO: Ben Sackey **Location: 5b31 / 5c18** **Art Unit: 1626** **Tuesday, August 23, 2005** **Case Serial Number: 10/631268**	**From: Noble Jarrell** **Location: Biotech-Chem Library** **Rem 1B71** **Phone: 272-2556** **Noble.jarrell@uspto.gov**

Search Notes

Noble

Access DB# 161714

SEARCH REQUEST FORM

Scientific and Technical Information Center

Requester's Full Name: BEN SACKEY Examiner #: 73489 Date: 8/6/05
Art Unit: 1626 Phone Number ~~30~~ 2-0704 Serial Number: 10/631,268
Mail Box and Bldg/Room Location: REM 5B31 Results Format Preferred (circle): PAPER DISK E-MAIL

If more than one search is submitted, please prioritize searches in order of need.

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Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc, if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

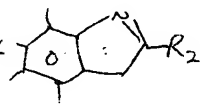
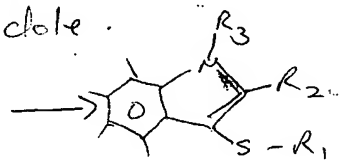
Title of Invention: 3-Sulfenylation of indole-2-carboxylates

Inventors (please provide full names): Hamilton et al.

Earliest Priority Filing Date: 7/31/02

**For Sequence Searches Only* Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.*

A one-step method of sulfenylation of 2-carboxyindoles comprising mixing N-chlorosuccinimide and R_1SH, combining and indole carboxylate with a mixture containing sulfenyl chloride to produce sulfenylated indole.

$NCS + R_1SH \longrightarrow R_1SCl +$  

Thanks

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Searcher: noble	NA Sequence (#)		STN
Searcher Phone #:	AA Sequence (#)		Dialog
Searcher Location:	Structure (#)	4/	Questel/Orbit
Date Searcher Picked Up:	Bibliographic	1	Dr.Link
Date Completed: 8/23/05	Litigation		Lexis/Nexis
Searcher Prep & Review Time: 5310	Fulltext		Sequence Systems
Clerical Prep Time:	Patent Family		WWW/Internet
Online Time: 53	Other		Other (specify)

PTO-1590 (8-01)

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     FILE 'HCAPLUS' ENTERED AT 09:40:51 ON 23 AUG 2005
L1            1 US2004133014/PN OR (US2003-631268# OR US2002-400092#)/AP,PRN

     FILE 'REGISTRY' ENTERED AT 09:41:42 ON 23 AUG 2005

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L2              TRA L1 1- RN :       42 TERMS

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L3           42 SEA L2

     FILE 'WPIX' ENTERED AT 09:41:48 ON 23 AUG 2005
L4            1 L1

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FILE COVERS 1907 - 23 Aug 2005  VOL 143 ISS 9
FILE LAST UPDATED: 22 Aug 2005  (20050822/ED)

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 This file contains CAS Registry Numbers for easy and accurate
 substance identification.

=> d all l1 tot

L1   ANSWER 1 OF 1  HCAPLUS  COPYRIGHT 2005 ACS on STN
AN   2004:550803  HCAPLUS
DN   141:106369
ED   Entered STN:  09 Jul 2004
TI   Method for 3-sulfenylation of indole-2-carboxylates by chlorination of
     thiols to sulfenyl chlorides
IN   Wall Hamilton, Harriet; Krasutsky, Alexei P.; Reed, Jessica; Schlosser,
     Kevin
PA   USA
SO   U.S. Pat. Appl. Publ., 13 pp.
     CODEN: USXXCO
DT   Patent
LA   English
IC   ICM  C07D209-36
INCL 548484000
CC   27-11 (Heterocyclic Compounds (One Hetero Atom))
FAN.CNT 1
     PATENT NO.          KIND   DATE        APPLICATION NO.        DATE
     ---------------     ----   --------    --------------------   --------
PI   US 2004133014        A1    20040708    US 2003-631268         20030731 <--
PRAI US 2002-400092P      P     20020731  <--
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Search done by ~~Rose Sabine~~

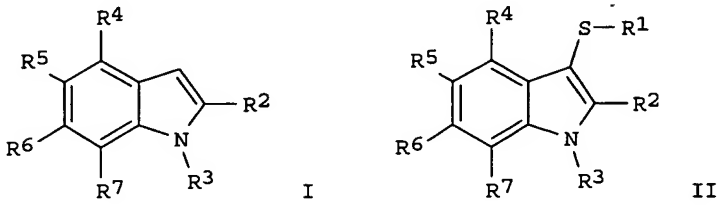
Noble Jarrell

CLASS

PATENT NO.	CLASS	PATENT FAMILY CLASSIFICATION CODES
US 2004133014	ICM	C07D209-36
	INCL	548484000
US 2004133014	NCL	548/484.000
	ECLA	C07D209/42; C07D513/04+281B+209B <--

OS CASREACT 141:106369; MARPAT 141:106369

GI



AB A highly efficient one-pot procedure for 3-sulfenylation of indole 2-carboxylates is described. Treatment of thiols of formula HSR1 [R1 = C1-6 alkyl, C2-6 alkoxycarbonyl, C3-7 cycloalkyl, C3-7 heterocycloalkyl, C1-6 alkyl-S(O)mRa, -C1-6 alkyl-S(O)mNRbRc, C1-6 alkyl-NRbRc, or C1-6 alkyl-CO-NRbRc, aryl, heteroaryl, etc.; m = 1, 2; Ra, Rb, Rc = H, C1-6 alkyl, C3-7 cycloalkyl, C3-6 heterocycloalkyl, or aryl] with N-chlorosuccinimide at -78° in CH2Cl2 affords sulfenyl chlorides of formula R1SCl in situ that readily react with indole 2-carboxylates [I; R2 = CO2H, tetrazolyl, C2-6 alkoxycarbonyl, CONRbRc (Rb, Rc = H, C1-6 alkyl), S(O)mRa, or -S(O)mNRbRc, NRbRc, etc. (wherein m = 1, 2; Ra, Rb, Rc = H, C1-6 alkyl, C3-7 cycloalkyl, C3-6 heterocycloalkyl, or aryl); R3 = H, each optionally substituted alkyl C1-6 or C1-6 alkanoyl; R4-R7 = H, halo, C1-6 alkyl, C1-6 alkoxy, cyano, C3-7 cycloalkyl, C3-7 heterocycloalkyl, C1-6 alkyl-S(O)mRa, -C1-6 -S(O)mNRbRc, C1-6 alkyl-NRbRc, or C1-6 alkyl-CO-NRbRc, C1-6 alkyl-COR1, S(O)mRa, S(O)mNRbRc, NRbRc, CONRbRc, aryl, heteroaryl, etc. (wherein m = 1, 2; Ra, Rb, Rc = H, C1-6 alkyl, C3-7 cycloalkyl, C3-6 heterocycloalkyl, heteroaryl or aryl)] to give 3-thioindoles (II) in high yields. This new method is milder, produces less waste, and is compatible with a wide range of thiol and indole functionality. It is also used for intramol. sulfenylation of N-(2-mercaptoethyl)-1H-indolecarboxamide derivs. to form a indole-fused 1,4-thiaazepane ring. Thus, to a cooled solution of N-chlorosuccinimide (2.74 g, 20.6 mmol) in CH2Cl2 (125 mL) at -78°, 3-methoxythiophenol (2.55 mL, 20.6 mmol) was added. The reaction was warmed to 0° over 15 min and a solution of indole-2-carboxylic acid Me ester (3 g, 17.1 mmol) in CH2Cl2 (25 mL) was added. The reaction stirred at 0° for 1 h, then concentrated under reduced pressure and the residue was suspended in H2O and stirred for 30 min to give, after filtration of the solid and recrystn. from EtOAc/hexanes to give 3-[(3-methoxyphenyl)thio]-1H-indole-2-carboxylic acid Me ester (3.22 g, 60%).

ST phenylthioindolecarboxylate alkylthioindolecarboxylate prepn; chlorosuccinimide chlorination thiol; thiol chlorination sulfenylation indolecarboxylate; sulfenyl chloride prepn sulfenylation indolecarboxylate; intramol sulfenylation mercaptoethylindolecarboxamide indolothiaazepane prepn

IT Chlorination
(3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

IT Thiols, reactions
RL: RCT (Reactant); RACT (Reactant or reagent)
(3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

IT Cyclization
(intramol. sulfenylation of N-(2-mercaptoethyl)indolecarboxamide

derivs. to indole-fused 1,4-thiaazacycloheptane ring; 3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

IT Sulfenyl compounds
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(sulfenyl chlorides; 3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

IT Substitution reaction
(sulfenylation; 3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

IT 60-23-1, 2-Aminoethanethiol 75-66-1, tert-Butyl mercaptan 100-53-8, Benzyl mercaptan 106-45-6, 4-Methylthiophenol 106-53-6, 4-Bromothiophenol 108-40-7, 3-Methylthiophenol 108-98-5, Thiophenol, reactions 128-09-6, N-Chlorosuccinimide 348-36-7, 5-Fluoro-1H-indole-2-carboxylic acid ethyl ester 1202-04-6, 1H-Indole-2-carboxylic acid methyl ester 2935-90-2, 3-Mercaptopropanoic acid methyl ester 3770-50-1, 1H-Indole-2-carboxylic acid ethyl ester 4382-54-1, 5-Methoxy-1H-indole-2-carboxylic acid 6320-01-0, 3-Bromothiophenol 7217-59-6, 2-Methoxythiophenol 7684-18-6, 3-Amino-2-methylpropane-2-thiol 15570-12-4, 3-Methoxythiophenol 17481-19-5, 3-Chloropropanethiol 67385-09-5, 2-(tert-Butoxycarbonylamino)ethanethiol 67929-86-6, 5-Methoxy-1H-indole-2-carboxylic acid methyl ester 67929-87-7, 5-Methoxy-1-methyl-1H-indole-2-carboxylic acid methyl ester 130445-25-9, 5-Methoxy-1-methyl-1H-indole-2-carboxamide
RL: RCT (Reactant); RACT (Reactant or reagent)
(3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

IT 671783-58-7P, N-(2-Mercaptoethyl)5-methoxy-1H-indole-2-carboxamide 671783-60-1P, N-(2-Mercapto-2-methylpropyl)5-methoxy-1H-indole-2-carboxamide
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

IT 671783-29-2P, 5-Methoxy-1-methyl-3-[(3-chloropropyl)thio]-1H-indole-2-carboxylic acid methyl ester 671783-31-6P, 5-Methoxy-1-methyl-3-(phenylthio)-1H-indole-2-carboxylic acid methyl ester 671783-33-8P, 5-Methoxy-1-methyl-3-[(4-methylphenyl)thio]-1H-indole-2-carboxamide 671783-35-0P, 3-(Benzylthio)-5-methoxy-1-methyl-1H-indole-2-carboxamide 671783-42-9P, 5-Methoxy-3-[[2-(methoxycarbonyl)ethyl]thio]-1H-indole-2-carboxylic acid methyl ester 671783-44-1P, 5-Methoxy-3-[[2-(tert-butoxycarbonylamino)ethyl]thio]-1H-indole-2-carboxylic acid methyl ester 671783-46-3P, 3-[(3-Methylphenyl)thio]-1H-indole-2-carboxylic acid ethyl ester 671783-48-5P, 3-[(3-Methoxyphenyl)thio]-1H-indole-2-carboxylic acid ethyl ester 671783-50-9P, 3-[(3-Bromophenyl)thio]-1H-indole-2-carboxylic acid ethyl ester 671783-62-3P 671783-64-5P 718614-74-5P, 3-[(4-Bromophenyl)thio]-1H-indole-2-carboxylic acid ethyl ester 718614-75-6P, 3-[(2-Methoxyphenyl)thio]-1H-indole-2-carboxylic acid ethyl ester 718614-76-7P, 3-[(3-Methoxyphenyl)thio]-1H-indole-2-carboxylic acid methyl ester 718614-77-8P, 3-[(4-Bromophenyl)thio]-1H-indole-2-carboxylic acid methyl ester 718614-78-9P, 3-[(3-Methylphenyl)thio]-1H-indole-2-carboxylic acid methyl ester
RL: SPN (Synthetic preparation); PREP (Preparation)
(3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

IT 718614-72-3P, 5-Methoxy-1-methyl-3-(tert-butylthio)-1H-indole-2-carboxylic acid methyl ester 718614-73-4P, 5-Methoxy-1-methyl-3-(tert-butylthio)-1H-indole-2-carboxamide
RL: SPN (Synthetic preparation); PREP (Preparation)
(failed reaction; 3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

=> b wpix
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Search done by Ross Schipe

FILE LAST UPDATED: 18 AUG 2005 <20050818/UP>
MOST RECENT DERWENT UPDATE: 200553 <200553/DW>
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=> d all l4 tot

L4 ANSWER 1 OF 1 WPIX COPYRIGHT 2005 THE THOMSON CORP on STN
AN 2004-524924 [50] WPIX
DNC C2004-193108
TI Preparation of 3-thioindole-2-carboxylate derivatives useful in treatment of e.g. HIV, obesity involves sulfenylation of 2-carboxy-indole derivatives with in situ generated sulfenyl chloride.
DC B02
IN KRASUTSKY, A P; REED, J; SCHLOSSER, K; WALL HAMILTON, H
PA (KRAS-I) KRASUTSKY A P; (REED-I) REED J; (SCHL-I) SCHLOSSER K; (HAMI-I) WALL HAMILTON H
CYC 1
PI US 2004133014 A1 20040708 (200450)* 13 C07D209-36 <--
ADT US 2004133014 A1 Provisional US 2002-400092P 20020731, US 2003-631268 20030731
PRAI US 2002-400092P 20020731; US 2003-631268 20030731
IC ICM C07D209-36
AB US2004133014 A UPAB: 20040805
NOVELTY - Sulfenylation of 2-carboxy-indole derivatives involves reacting N-chlorosuccinimide with thiols to generate sulfenyl chloride, which is reacted with indole-2-carboxylate derivatives to give 3-sulfenylated-2-carboxy-indole derivatives.
DETAILED DESCRIPTION - Sulfenylation (M1) of 2-carboxy-indole derivatives of formula (I) involves (a) mixing N-chlorosuccinimide with thiol in a liquid (L1) to form sulfenyl chloride of formula R1SCl as a homogeneous or heterogeneous mixture and (b) combining the in situ generated sulfenyl chloride with (I) to give 3-sulfenylated indole derivatives of formula (II).
R1 = 1-6C alkyl, 3-7C (hetero)cycloalkyl (all optionally partially unsaturated and substituted by T1 or aryl(1-6C) alkoxy), (hetero)aryl (optionally substituted by T1 and aryl(1-6C)alkoxy), 2-6C alkoxycarbonyl, (1-6C)-S(O)mRa, -(1-6C)-S(O)mNRbRc, (1-6C)-NRbRc or (1-6C)-C(=O)-NRbRc;
T1 = T2, arylcarbonyl, 1-6C alkoxy, 1-6C alkanoyl, 1-6C alkoxycarbonyl, 1-6C alkanoyloxy, -S(O)mRa, -S(O)mNRbRc, NRbRc or -C(=O)NRbRc;

T2 = (hetero)aryl, (hetero)aryloxy, hydroxy, nitro, halo or cyano;
m = 1 or 2;
Ra, Rb, and Rc = H, 1-6C alkyl, 3-7C (hetero)cycloalkyl or aryl;
R2 = carboxy, tetrazolyl, 2-6C alkoxycarbonyl, -C(O)NRb''Rc'', -S(O)mRa, -S(O)mNRb''Rc'', NRb''Rc'' or -C(=O)Rd (optionally substituted by T2);
Rb'' and Rc'' = H or 1-6C alkyl;
R3 = H, 1-6C alkyl or 1-6C alkanoyl (optionally substituted by T2);
R4 - R7 = 1-6C alkyl, 3-7C (hetero)cycloalkyl (all optionally partially unsaturated and substituted by T1) or (hetero)aryl (optionally substituted by T1), H, halo, 1-6C alkoxy, cyano, (1-6C)-S(O)mRa, -(1-6C)-S(O)mNRb'Rc', (1-6C)-NRb'Rc', (1-6C)-C(=O)-NRb'Rc', (1-6C)-C(=O)R1, S(O)mRa', S(O)mNRb'Rc', NRb'Rc', C(=O)-NRb'Rc' or C(=O)Rd;
Ra', Rb' and Rc' = Ra or heteroaryl.
Provided that not all of R4 - R7 are H.
NB: Rd not defined.
An INDEPENDENT CLAIM is included for intramolecular sulfenylation (M2) of 2-carboxyindoles involving mixing N-chlorosuccinimide with a thiol of formula (III) in liquid (L1) to generate sulfenyl chloride of formula (IV) followed by intramolecular sulfenylation of (IV) to give sulfenylated indole derivative of formula (V).
R8 and R9 = H or 1-6C alkyl (optionally substituted by T2);
n = 0 - 4;
X = CR7R8, O or NRb' where Rb' = H, acyl or 1-6C alkyl (optionally substituted with T2).
ACTIVITY - Anti-HIV; Anorectic; Antiallergic.
MECHANISM OF ACTION - Endothelial antagonist.
USE - For the preparation of 3-thioindole-2-carboxylates (claimed), which are useful as endothelin antagonists, anti-allergy agents and also in the treatment of HIV and obesity.
ADVANTAGE - The method is one step and includes milder conditions than those associated with the use of corrosive chlorine or sulfuryl chloride, as well as fast reaction times, easy work-up and improved yields. The in-situ formation method using N-chlorosuccinimide enhances the scope of the reaction, previously limited by the stability and ease of isolation of sulfenyl chlorides, avoids the formation of one equivalent of wasted thiol that occurs when a disulfide is used as the electrophilic sulfur source and possesses greater flexibility than other reported methods because the indole nitrogen does not require protection.
Dwg.0/0
FS CPI
FA AB; GI; DCN
MC CPI: B06-H; B14-A02B1; B14-E12; B14-G02A; B14-L06

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DICTIONARY FILE UPDATES:  22 AUG 2005  HIGHEST RN 861291-85-2

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L25             STR
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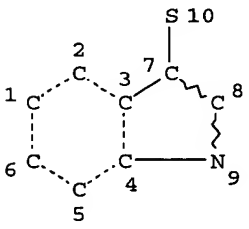


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STEREO ATTRIBUTES: NONE
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Noble Jarrell

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L44 ANSWER 1 OF 9 REGISTRY COPYRIGHT 2005 ACS on STN
RN 815597-82-1 REGISTRY
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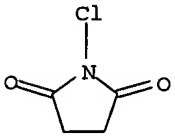
LC STN Files: CA, CAPLUS
CRN (128-09-6)



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1 REFERENCES IN FILE CA (1907 TO DATE)
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L44 ANSWER 2 OF 9 REGISTRY COPYRIGHT 2005 ACS on STN
RN 815597-81-0 REGISTRY
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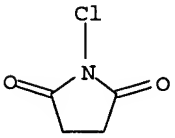
LC STN Files: CA, CAPLUS
CRN (128-09-6)



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L44 ANSWER 3 OF 9 REGISTRY COPYRIGHT 2005 ACS on STN
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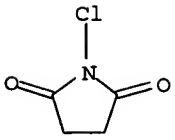

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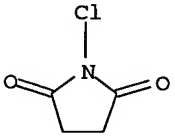
1 REFERENCES IN FILE CA (1907 TO DATE)
1 REFERENCES IN FILE CAPLUS (1907 TO DATE)

L44 ANSWER 4 OF 9 REGISTRY COPYRIGHT 2005 ACS on STN
RN **90685-90-8** REGISTRY
ED Entered STN: 16 Nov 1984
CN 2,5-Pyrrolidinedione, 1-chloro-, monoammoniate (9CI) (CA INDEX NAME)
MF **C4 H4 Cl N O2 . H3 N**
LC STN Files: CA, CAPLUS
CRN (128-09-6)



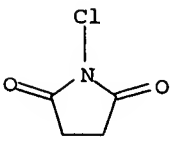
● NH_3

1 REFERENCES IN FILE CA (1907 TO DATE)
1 REFERENCES IN FILE CAPLUS (1907 TO DATE)

L44 ANSWER 5 OF 9 REGISTRY COPYRIGHT 2005 ACS on STN
RN **90124-80-4** REGISTRY
ED Entered STN: 16 Nov 1984
CN 2,5-Pyrrolidinedione, 1-chloro-, compd. with thiobis[methane] (1:1) (9CI)
(CA INDEX NAME)
OTHER CA INDEX NAMES:
CN Methane, thiobis-, compd. with 1-chloro-2,5-pyrrolidinedione (1:1) (9CI)
MF **C4 H4 Cl N O2 . C2 H6 S**
LC STN Files: BEILSTEIN*, CA, CAPLUS, CASREACT
(*File contains numerically searchable property data)

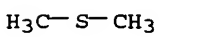
CM 1

CRN 128-09-6
CMF C4 H4 Cl N O2



CM 2

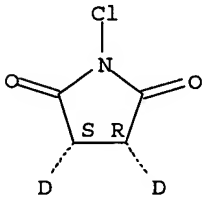
CRN 75-18-3
CMF C2 H6 S



1 REFERENCES IN FILE CA (1907 TO DATE)
1 REFERENCES IN FILE CAPLUS (1907 TO DATE)

L44 ANSWER 6 OF 9 REGISTRY COPYRIGHT 2005 ACS on STN
RN **66996-79-0** REGISTRY
ED Entered STN: 16 Nov 1984
CN 2,5-Pyrrolidinedione-3,4-d2, 1-chloro-, cis- (9CI) (CA INDEX NAME)
FS STEREOSEARCH
MF **C4 H2 Cl D2 N O2**
LC STN Files: CA, CAPLUS

Relative stereochemistry.

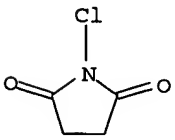


2 REFERENCES IN FILE CA (1907 TO DATE)
2 REFERENCES IN FILE CAPLUS (1907 TO DATE)

L44 ANSWER 7 OF 9 REGISTRY COPYRIGHT 2005 ACS on STN
RN **54437-73-9** REGISTRY
ED Entered STN: 16 Nov 1984
CN 2,5-Pyrrolidinedione, 1-chloro-, compd. with N,N-diethylethanamine (1:1) (9CI) (CA INDEX NAME)
OTHER CA INDEX NAMES:
CN Ethanamine, N,N-diethyl-, compd. with 1-chloro-2,5-pyrrolidinedione (1:1)
MF **C6 H15 N . C4 H4 Cl N O2**
LC STN Files: CA, CAPLUS

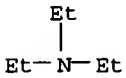
CM 1

CRN 128-09-6
CMF C4 H4 Cl N O2



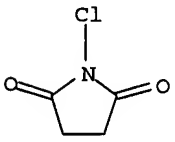
CM 2

CRN 121-44-8
CMF C6 H15 N



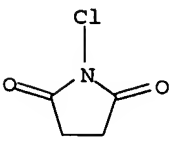
1 REFERENCES IN FILE CA (1907 TO DATE)
1 REFERENCES IN FILE CAPLUS (1907 TO DATE)

L44 ANSWER 8 OF 9 REGISTRY COPYRIGHT 2005 ACS on STN
RN **38144-43-3** REGISTRY
ED Entered STN: 16 Nov 1984
CN 2,5-Pyrrolidinedione, 1-chloro-, radical ion(1-) (9CI) (CA INDEX NAME)
OTHER NAMES:
CN N-Chlorosuccinimide anion radical
MF **C4 H4 Cl N O2**
CI RIS
LC STN Files: CA, CAPLUS



5 REFERENCES IN FILE CA (1907 TO DATE)
5 REFERENCES IN FILE CAPLUS (1907 TO DATE)

L44 ANSWER 9 OF 9 REGISTRY COPYRIGHT 2005 ACS on STN
RN **128-09-6** REGISTRY
ED Entered STN: 16 Nov 1984
CN 2,5-Pyrrolidinedione, 1-chloro- (9CI) (CA INDEX NAME)
OTHER CA INDEX NAMES:
CN Succinimide, N-chloro- (6CI, 7CI, 8CI)
OTHER NAMES:
CN 1-Chloro-2,5-pyrrolidinedione
CN Chlorosuccinimide
CN N-Chlorosuccinimide
CN NSC 8748
CN Succinchlorimide
CN Succinic N-chloroimide
FS 3D CONCORD
MF **C4 H4 Cl N O2**
CI COM
LC STN Files: AGRICOLA, ANABSTR, BEILSTEIN*, BIOBUSINESS, BIOSIS, CA, CANCERLIT, CAOLD, CAPLUS, CASREACT, CBNB, CEN, CHEMCATS, CHEMINFORMRX, CHEMLIST, CIN, CSCHEM, DETHERM*, EMBASE, GMELIN*, HODOC*, HSDB*, IFICDB, IFIPAT, IFIUDB, IPA, MEDLINE, MRCK*, MSDS-OHS, PIRA, PROMT, PS, RTECS*, SPECINFO, SYNTHLINE, TOXCENTER, USAN, USPAT2, USPATFULL
(*File contains numerically searchable property data)
Other Sources: DSL**, EINECS**, TSCA**
(**Enter CHEMLIST File for up-to-date regulatory information)



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

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          1261 REFERENCES IN FILE CA (1907 TO DATE)
            14 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
          1272 REFERENCES IN FILE CAPLUS (1907 TO DATE)
            41 REFERENCES IN FILE CAOLD (PRIOR TO 1967)


=> d his

     (FILE 'HOME' ENTERED AT 09:40:45 ON 23 AUG 2005)

     FILE 'HCAPLUS' ENTERED AT 09:40:51 ON 23 AUG 2005
L1           1 US2004133014/PN OR (US2003-631268# OR US2002-400092#)/AP,PRN

     FILE 'REGISTRY' ENTERED AT 09:41:42 ON 23 AUG 2005

     FILE 'HCAPLUS' ENTERED AT 09:41:42 ON 23 AUG 2005
L2             TRA L1 1- RN :       42 TERMS

     FILE 'REGISTRY' ENTERED AT 09:41:43 ON 23 AUG 2005
L3          42 SEA L2

     FILE 'WPIX' ENTERED AT 09:41:48 ON 23 AUG 2005
L4           1 L1

     FILE 'HCAPLUS' ENTERED AT 09:50:40 ON 23 AUG 2005
                E WALL/AU
                E WALL H/AU
L5           7 E3
                E WALL HAMILTON/AU
L6           1 E4
                E HAMILTON H/AU
L7          28 E3-4,E23
                E HAMILTON HARRIET/AU
L8          46 E3-6
                E KRASUTSKY A/AU
L9           9 E4-6
                E REED J/AU
L10         72 E3-4
                E REED JESS/AU
L11         56 E3-9
                E SCHLOSSER K/AU
L12         15 E3,E8-9
                E WRANER/CS,PA
                E WARNNER/CS,PA
                E WARNER/CS,PA
L13       7002 E3-4
                E WARNERLAMB/CS,PA
L14          5 E5-7
                E WARNER LAMB/CS,PA
L15       5749 WARNER LAMB?/CS,PA
                E SUBSTITUTION REAC/CT
                E E4+ALL
L16        309 SUBSTITUTION REACTION+OLD,NT/CT (L)(SULFEN? OR SULPHEN?)
                E SULFENYL COMPOUNDS/CT
                E E3+ALL
L17       9296 SULFENYL COMPOUNDS+NT/CT
L18        937 L17 (L) (?FLUORIDE? OR ?CHLORIDE? OR ?BROMID? OR ?IODID? OR ?HA
L19          3 L18 (L) PREP+NT/RL AND L16
L20          1 L19 AND L5-15
L21          2 L19 NOT L20
L22            QUE PY<=2002 OR AY<=2002 OR PRY<=2002 OR PD<20020731 OR AD<2002
L23          2 L21 AND L22
L24          2 L21,L23

     FILE 'REGISTRY' ENTERED AT 10:00:02 ON 23 AUG 2005
L25            STR
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L26          50 L25
L27        4263 L25 FULL

     FILE 'HCAPLUS' ENTERED AT 10:01:59 ON 23 AUG 2005
L28         820 L27
L29         515 L28 (L) PREP+NT/RL
L30          51 L17 AND L16
L31           1 L30 AND L5-15
L32          50 L30 NOT L31
                E THIOL/CT
                E THIOLS/CT
                E E3+ALL
                E E2
                E E3+ALL
L33             QUE "THIOLS (ORGANIC)"+OLD,NT/CT
L34           6 L33 AND L32
L35          11 ?THIOL? AND L32
L36          11 L34,L35
L37       42909 L33 (L) RACT+NT/RL
L38       37627 ?THIOL? (L) RACT+NT/RL
L39           9 L37,L38 AND L36
L40          11 L36,L39

     FILE 'REGISTRY' ENTERED AT 10:05:41 ON 23 AUG 2005
L41           1 128-09-6
L42          22 C4H4CLNO2 AND NC4/ES
L43          14 L42 NOT (MXS/CI OR MIXT)
                SEL RN 1-3 7-9 11 13-14 L43
L44           9 E1-9 AND L43
L45           5 L43 NOT L44
L46           8 L42 NOT L43
L47          13 L45,L46

     FILE 'HCAPLUS' ENTERED AT 10:10:30 ON 23 AUG 2005
L48        1276 L44
L49        2654 2(1A)5(1A)(PYRROLIDINEDIONE? OR PYRROLIDIN?(1A)DIONE?) (4A)CHLO
L50        1117 L48,L49(L) RACT+NT/RL
L51           1 L20,L31 AND L50
L52           1 L20,L31,L51
L53           0 L24,L40 AND L48-50
L54          12 L24,L40

     FILE 'CASREACT' ENTERED AT 10:17:15 ON 23 AUG 2005
L55             STR
L56           0 L55
L57          12 L55 FULL
                E HAILTON H/AU
                E HAMILTON H/AU
L58          16 E5-7
                E HAMILTON WALL/AU
                E KRASULSKY A/AU
                E SCHLOSSER K/AU
L59           2 E4-5
                E REED J/AU
L60          19 E3-7,E10-11
L61         644 (WARNERLAMB? OR WARNER LAMB?)/CS,PA
L62           0 L57 AND L58-61
L63          31 L58-61 AND ?THIOL?
L64           4 L58-60 AND ?THIOL?
                SEL AN 1-2 L64
L65           2 E1-2 AND L64
L66          11 L57 AND L22
L67          12 L57,L66
L68           3 L63 AND SULFEN?
                SEL AN 2-3 5-12 L57
L69          10 E3-12 AND L57 AND L67
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Search done by Ross Schipe

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L70             STR
L71           0 L70
L72           7 L70 FULL
L73           0 L72 AND L58-61
L74           7 L72 AND L22
L75           7 L72,L74
L76           0 L69 AND L75
L77          17 L69,L75
L78           3 L65,L68

=> b hcap
FILE 'HCAPLUS' ENTERED AT 10:39:42 ON 23 AUG 2005
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
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FILE COVERS 1907 - 23 Aug 2005  VOL 143 ISS 9
FILE LAST UPDATED: 22 Aug 2005  (20050822/ED)

New CAS Information Use Policies, enter HELP USAGETERMS for details.

 This file contains CAS Registry Numbers for easy and accurate
 substance identification.

=> d all fhitstr l52 tot

L52  ANSWER 1 OF 1  HCAPLUS  COPYRIGHT 2005 ACS on STN
AN   2004:550803  HCAPLUS
DN   141:106369
ED   Entered STN:  09 Jul 2004
TI   Method for 3-sulfenylation of indole-2-carboxylates by chlorination of
     thiols to sulfenyl chlorides
IN   Wall Hamilton, Harriet; Krasutsky, Alexei P.;
     Reed, Jessica; Schlosser, Kevin
PA   USA
SO   U.S. Pat. Appl. Publ., 13 pp.
     CODEN: USXXCO
DT   Patent
LA   English
IC   ICM  C07D209-36
INCL 548484000
CC   27-11 (Heterocyclic Compounds (One Hetero Atom))
FAN.CNT 1
     PATENT NO.          KIND   DATE        APPLICATION NO.        DATE
     --------------      ----   --------    --------------------   --------
PI   US 2004133014        A1    20040708    US 2003-631268         20030731
PRAI US 2002-400092P      P     20020731
CLASS
 PATENT NO.       CLASS  PATENT FAMILY CLASSIFICATION CODES
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 US 2004133014    ICM    C07D209-36
                  INCL   548484000
 US 2004133014    NCL    548/484.000
                  ECLA   C07D209/42; C07D513/04+281B+209B
OS   CASREACT 141:106369; MARPAT 141:106369
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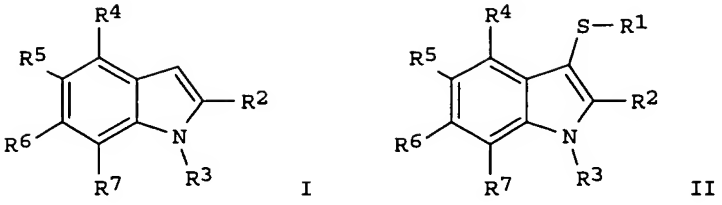
GI



I II

AB A highly efficient one-pot procedure for 3-sulfenylation of indole 2-carboxylates is described. Treatment of thiols of formula HSR1 [R1 = C1-6 alkyl, C2-6 alkoxycarbonyl, C3-7 cycloalkyl, C3-7 heterocycloalkyl, C1-6 alkyl-S(O)mRa, -C1-6 alkyl-S(O)mNRbRc, C1-6 alkyl-NRbRc, or C1-6 alkyl-CO-NRbRc, aryl, heteroaryl, etc.; m = 1, 2; Ra, Rb, Rc = H, C1-6 alkyl, C3-7 cycloalkyl, C3-6 heterocycloalkyl, or aryl] with N-chlorosuccinimide at -78° in CH2Cl2 affords sulfenyl chlorides of formula R1SCl in situ that readily react with indole 2-carboxylates [I; R2 = CO2H, tetrazolyl, C2-6 alkoxycarbonyl, CONRbRc (Rb, Rc = H, C1-6 alkyl), S(O)mRa, or -S(O)mNRbRc, NRbRc, etc. (wherein m = 1, 2; Ra, Rb, Rc = H, C1-6 alkyl, C3-7 cycloalkyl, C3-6 heterocycloalkyl, or aryl); R3 = H, each optionally substituted alkyl C1-6 or C1-6 alkanoyl; R4-R7 = H, halo, C1-6 alkyl, C1-6 alkoxy, cyano, C3-7 cycloalkyl, C3-7 heterocycloalkyl, C1-6 alkyl-S(O)mRa, -C1-6 -S(O)mNRbRc, C1-6 alkyl-NRbRc, or C1-6 alkyl-CO-NRbRc, C1-6 alkyl-COR1, S(O)mRa, S(O)mNRbRc, NRbRc, CONRbRc, aryl, heteroaryl, etc. (wherein m = 1, 2; Ra, Rb, Rc = H, C1-6 alkyl, C3-7 cycloalkyl, C3-6 heterocycloalkyl, heteroaryl or aryl)] to give 3-thioindoles (II) in high yields. This new method is milder, produces less waste, and is compatible with a wide range of thiol and indole functionality. It is also used for intramol. sulfenylation of N-(2-mercaptoethyl)-1H-indolecarboxamide derivs. to form a indole-fused 1,4-thiaazepane ring. Thus, to a cooled solution of N-chlorosuccinimide (2.74 g, 20.6 mmol) in CH2Cl2 (125 mL) at -78°, 3-methoxythiophenol (2.55 mL, 20.6 mmol) was added. The reaction was warmed to 0° over 15 min and a solution of indole-2-carboxylic acid Me ester (3 g, 17.1 mmol) in CH2Cl2 (25 mL) was added. The reaction stirred at 0° for 1 h, then concentrated under reduced pressure and the residue was suspended in H2O and stirred for 30 min to give, after filtration of the solid and recrystn. from EtOAc/hexanes to give 3-[(3-methoxyphenyl)thio]-1H-indole-2-carboxylic acid Me ester (3.22 g, 60%).

ST phenylthioindolecarboxylate alkylthioindolecarboxylate prepn; chlorosuccinimide chlorination thiol; thiol chlorination sulfenylation indolecarboxylate; sulfenyl chloride prepn sulfenylation indolecarboxylate; intramol sulfenylation mercaptoethylindolecarboxamide indolothiaazepane prepn

IT Chlorination
(3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

IT Thiols, reactions
RL: RCT (Reactant); RACT (Reactant or reagent)
(3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

IT Cyclization
(intramol. sulfenylation of N-(2-mercaptoethyl)indolecarboxamide derivs. to indole-fused 1,4-thiaazacycloheptane ring; 3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

IT **Sulfenyl compounds**
RL: RCT (Reactant); **SPN (Synthetic preparation); PREP (Preparation)**; RACT (Reactant or reagent)

(sulfenyl chlorides; 3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

IT **Substitution reaction**
(sulfenylation; 3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

IT 60-23-1, 2-Aminoethanethiol 75-66-1, tert-Butyl mercaptan 100-53-8, Benzyl mercaptan 106-45-6, 4-Methylthiophenol 106-53-6, 4-Bromothiophenol 108-40-7, 3-Methylthiophenol 108-98-5, Thiophenol, reactions **128-09-6, N-Chlorosuccinimide** 348-36-7, 5-Fluoro-1H-indole-2-carboxylic acid ethyl ester 1202-04-6, 1H-Indole-2-carboxylic acid methyl ester 2935-90-2, 3-Mercaptopropanoic acid methyl ester 3770-50-1, 1H-Indole-2-carboxylic acid ethyl ester 4382-54-1, 5-Methoxy-1H-indole-2-carboxylic acid 6320-01-0, 3-Bromothiophenol 7217-59-6, 2-Methoxythiophenol 7684-18-6, 3-Amino-2-methylpropane-2-thiol 15570-12-4, 3-Methoxythiophenol 17481-19-5, 3-Chloropropanethiol 67385-09-5, 2-(tert-Butoxycarbonylamino)ethanethiol 67929-86-6, 5-Methoxy-1H-indole-2-carboxylic acid methyl ester 67929-87-7, 5-Methoxy-1-methyl-1H-indole-2-carboxylic acid methyl ester 130445-25-9, 5-Methoxy-1-methyl-1H-indole-2-carboxamide
RL: **RCT (Reactant); RACT (Reactant or reagent)**
(3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

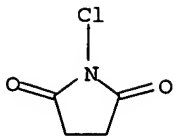
IT 671783-58-7P, N-(2-Mercaptoethyl)5-methoxy-1H-indole-2-carboxamide 671783-60-1P, N-(2-Mercapto-2-methylpropyl)5-methoxy-1H-indole-2-carboxamide
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

IT 671783-29-2P, 5-Methoxy-1-methyl-3-[(3-chloropropyl)thio]-1H-indole-2-carboxylic acid methyl ester 671783-31-6P, 5-Methoxy-1-methyl-3-(phenylthio)-1H-indole-2-carboxylic acid methyl ester 671783-33-8P, 5-Methoxy-1-methyl-3-[(4-methylphenyl)thio]-1H-indole-2-carboxamide 671783-35-0P, 3-(Benzylthio)-5-methoxy-1-methyl-1H-indole-2-carboxamide 671783-42-9P, 5-Methoxy-3-[[2-(methoxycarbonyl)ethyl]thio]-1H-indole-2-carboxylic acid methyl ester 671783-44-1P, 5-Methoxy-3-[[2-(tert-butoxycarbonylamino)ethyl]thio]-1H-indole-2-carboxylic acid methyl ester 671783-46-3P, 3-[(3-Methylphenyl)thio]-1H-indole-2-carboxylic acid ethyl ester 671783-48-5P, 3-[(3-Methoxyphenyl)thio]-1H-indole-2-carboxylic acid ethyl ester 671783-50-9P, 3-[(3-Bromophenyl)thio]-1H-indole-2-carboxylic acid ethyl ester 671783-62-3P 671783-64-5P 718614-74-5P, 3-[(4-Bromophenyl)thio]-1H-indole-2-carboxylic acid ethyl ester 718614-75-6P, 3-[(2-Methoxyphenyl)thio]-1H-indole-2-carboxylic acid ethyl ester 718614-76-7P, 3-[(3-Methoxyphenyl)thio]-1H-indole-2-carboxylic acid methyl ester 718614-77-8P, 3-[(4-Bromophenyl)thio]-1H-indole-2-carboxylic acid methyl ester 718614-78-9P, 3-[(3-Methylphenyl)thio]-1H-indole-2-carboxylic acid methyl ester
RL: SPN (Synthetic preparation); PREP (Preparation)
(3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

IT 718614-72-3P, 5-Methoxy-1-methyl-3-(tert-butylthio)-1H-indole-2-carboxylic acid methyl ester 718614-73-4P, 5-Methoxy-1-methyl-3-(tert-butylthio)-1H-indole-2-carboxamide
RL: SPN (Synthetic preparation); PREP (Preparation)
(failed reaction; 3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

IT **128-09-6, N-Chlorosuccinimide**
RL: **RCT (Reactant); RACT (Reactant or reagent)**
(3-sulfenylation of indole-2-carboxylates via in situ formation of sulfenyl chlorides by chlorination of thiols)

RN 128-09-6 HCAPLUS
CN 2,5-Pyrrolidinedione, 1-chloro- (9CI) (CA INDEX NAME)



=> d all hitstr l54 tot

L54 ANSWER 1 OF 12 HCAPLUS COPYRIGHT 2005 ACS on STN
AN 2004:1153318 HCAPLUS
DN 142:176307
ED Entered STN: 30 Dec 2004
TI Reaction of **Thiols** with N-Bonded Sulfenamide Complexes of Cobalt(III): Steric Effect and Reaction Pathway
AU Sisley, Margaret J.; Ferguson, Michael J.; Jordan, Robert B.
CS Department of Chemistry and X-ray Crystallography Laboratory, University of Alberta, Edmonton, AB, T6G 2G2, Can.
SO Inorganic Chemistry (2005), 44(2), 293-299
CODEN: INOCAJ; ISSN: 0020-1669
PB American Chemical Society
DT Journal
LA English
CC 22-4 (Physical Organic Chemistry)
Section cross-reference(s): 67, 75
AB The products and kinetics of the reaction of several **thiols** (RSH = 2-**aminoethanethiol**, cysteine, penicillamine, cysteine Et ester) with N-bonded sulfenamide complexes ([Co(en)2(NH2S(CH2)2NH2)]3+ (IA), [Co(en)2(NH2SCH2CH(CO2H)NH2)]3+ (IC), [Co(en)2(NH2SC(CH3)2CH(CO2H)NH2)]3+ (IP)) have been studied. The reaction proceeds by nucleophilic attack at sulfur with cleavage of the N-S bond to form a disulfide and leave a coordinated NH3 ligand. The kinetics (pH 4-10) reveal that the deprotonated **thiol**, RS-, is the reactive nucleophile and that the N-deprotonated sulfenamide complex is unreactive. The reactions of IP are .apprx.104 times slower than those of IA or IC, and the reasons and consequences of this large steric effect are discussed. It is concluded, on the basis of these and other observations from the literature, that there will be substantial steric retardation to nucleophilic attack at two-coordinate sulfur in a R-C(CH3)2-S-X-R' unit because of the regiospecificity of the reaction. The acid dissociation consts. of IP and the X-ray structure of its bromide salt also are reported.
ST **thiol** reaction sulfenamide cobalt complex kinetics mechanism steric effect
IT **Substitution reaction, nucleophilic**
(at two-coordinate sulfur; steric effect on nucleophilic substitution reaction of **thiols** with N-bonded **sulfenamide** complexes of cobalt(III))
IT Ionization constant
(of Co sulfenamide complexes; steric effect on nucleophilic substitution reaction of **thiols** with N-bonded sulfenamide complexes of cobalt(III))
IT Crystal structure
Molecular structure
(of cobalt-sulfenamide complex derived from racemic penicillamine; steric effect on nucleophilic substitution reaction of **thiols** with N-bonded sulfenamide complexes of cobalt(III))
IT Reaction kinetics
Steric effects
(steric effect on nucleophilic substitution reaction of **thiols** with N-bonded sulfenamide complexes of cobalt(III))
IT **Thiols, reactions**
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); PRP (Properties); RCT (Reactant); PROC (Process);

RACT (Reactant or reagent)
(steric effect on nucleophilic substitution reaction of thiols with N-bonded sulfenamide complexes of cobalt(III))

IT Disulfides
RL: FMU (Formation, unclassified); FORM (Formation, nonpreparative)
(steric effect on nucleophilic substitution reaction of thiols with N-bonded sulfenamide complexes of cobalt(III))

IT Amides, reactions
Sulfenyl compounds
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); PRP (Properties); RCT (Reactant); PROC (Process);
RACT (Reactant or reagent)
(sulfenamides, Co complexes; steric effect on nucleophilic substitution reaction of thiols with N-bonded sulfenamide complexes of cobalt(III))

IT Exchange reaction
(thiol/disulfide; steric effect on nucleophilic substitution reaction of thiols with N-bonded sulfenamide complexes of cobalt(III))

IT 835596-65-1
RL: PRP (Properties)
(crystallog.; steric effect on nucleophilic substitution reaction of thiols with N-bonded sulfenamide complexes of cobalt(III))

IT 835596-67-3
RL: CPS (Chemical process); FMU (Formation, unclassified); PEP (Physical, engineering or chemical process); PRP (Properties); RCT (Reactant); FORM (Formation, nonpreparative); PROC (Process); RACT (Reactant or reagent)
(reduction kinetics; steric effect on nucleophilic substitution reaction of thiols with N-bonded sulfenamide complexes of cobalt(III))

IT 52-66-4 52-67-5, D-Penicillamine 52-90-4, L-Cysteine, reactions 60-23-1, 2-Aminoethanethiol 3411-58-3, L-Cysteine ethyl ester 754200-88-9 754200-93-6 754200-94-7
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); PRP (Properties); RCT (Reactant); PROC (Process);
RACT (Reactant or reagent)
(steric effect on nucleophilic substitution reaction of thiols with N-bonded sulfenamide complexes of cobalt(III))

IT 51-85-4, Cystamine 835596-66-2
RL: FMU (Formation, unclassified); FORM (Formation, nonpreparative)
(steric effect on nucleophilic substitution reaction of thiols with N-bonded sulfenamide complexes of cobalt(III))

RE.CNT 33 THERE ARE 33 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

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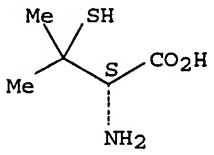
IT **52-67-5**, D-Penicillamine **52-90-4**, L-Cysteine, reactions
60-23-1, **2-Aminoethanethiol**
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); PRP (Properties); **RCT (Reactant)**; PROC (Process);
RACT (Reactant or reagent)
(steric effect on nucleophilic substitution reaction of **thiols** with N-bonded sulfenamide complexes of cobalt(III))

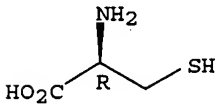
RN 52-67-5 HCAPLUS
CN D-Valine, 3-mercapto- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

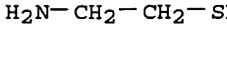


RN 52-90-4 HCAPLUS
CN L-Cysteine (9CI) (CA INDEX NAME)

Absolute stereochemistry.



RN 60-23-1 HCAPLUS
CN Ethanethiol, 2-amino- (8CI, 9CI) (CA INDEX NAME)



L54 ANSWER 2 OF 12 HCAPLUS COPYRIGHT 2005 ACS on STN
AN 2003:891789 HCAPLUS
DN 140:77215
ED Entered STN: 14 Nov 2003
TI Reaction of stable sulfenic and selenenic acids containing a bowl-type steric protection group with a phosphine. Elucidation of the mechanism of reduction of sulfenic and selenenic acids
AU Goto, Kei; Shimada, Keiichi; Nagahama, Michiko; Okazaki, Renji; Kawashima, Takayuki
CS Department of Chemistry, Graduate School of Science, The University of Tokyo, Tokyo, 113-0033, Japan
SO Chemistry Letters (2003), 32(11), 1080-1081
CODEN: CMLTAG; ISSN: 0366-7022
PB Chemical Society of Japan
DT Journal
LA English

CC 29-8 (Organometallic and Organometalloidal Compounds)
Section cross-reference(s): 22

AB The mechanism of the reduction of the stable sulfenic acid and selenenic acid containing a bowl-type steric protection group, 2,6-[2,6-(2,6-Me2C6H3)2C6H3CH2]2-4-tBuC6H2EOH (2,6-R2-4-tBuC6H2EOH, 1, 2, E = S, Se) by triphenylphosphine was examined by tracer studies using H218O. Oxidation of PPh3 by 1 and 2 in H218O (19.2% content 18O) produced 2,6-R2-4-tBuC6H2EH (4, 6, E = S and Se, resp.) and 18OPPh3; the isotope content in the oxide corresponds to 77% and 87% exchange rates, resp. These high exchange rates indicate, that the initial step of the reaction involves the attack of the phosphine on the sulfur or selenium atom of 1 and 2, resp., to give the 2,6-R2-4-tBuC6H2EP+Ph3 phosphonium intermediate which adds reversibly OH- from the reaction media and eliminates 4 or 6. This behavior contrasts with the reduction of tBuOOH by a phosphine, where the initial attack occurs on the hydroxylic oxygen.

ST sulfenic selenenic acid phosphine oxidn mechanism; **thiol** phosphine oxide formation mechanism sulfenic acid oxidn; selenol phosphine oxide formation mechanism selenenic acid oxidn

IT **Exchange reaction**
(isotope, oxygen-18; mechanism of triphenylphosphine oxidation by sterically protected **sulfenic** and selenenic acids, oxygen isotope exchange with water)

IT Selenenic acids
Sulfenic acids
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); RCT (Reactant); PROC (Process); RACT (Reactant or reagent)
(mechanism of triphenylphosphine oxidation by sterically protected sulfenic and selenenic acids)

IT Reduction
(mechanism; of sterically protected sulfenic and selenenic acids by triphenylphosphine, oxygen-18 isotope exchange)

IT Oxidation
(of triphenylphosphine by sterically protected sulfenic and selenenic acids, oxygen-18 isotope exchange)

IT 98511-61-6
RL: CPS (Chemical process); FMU (Formation, unclassified); PEP (Physical, engineering or chemical process); FORM (Formation, nonpreparative); PROC (Process)
(oxidation, oxygen isotope exchange; mechanism of triphenylphosphine oxidation by sterically protected sulfenic and selenenic acids, oxygen isotope exchange with water)

IT 603-35-0, Triphenylphosphine, reactions
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); RCT (Reactant); PROC (Process); RACT (Reactant or reagent)
(oxidation, oxygen isotope exchange; mechanism of triphenylphosphine oxidation by sterically protected sulfenic and selenenic acids, oxygen isotope exchange with water)

IT 177971-45-8 380305-90-8
RL: FMU (Formation, unclassified); FORM (Formation, nonpreparative)
(reduction product; mechanism of triphenylphosphine oxidation by sterically protected sulfenic and selenenic acids, oxygen isotope exchange with water)

IT 186256-53-1 380305-91-9
RL: RCT (Reactant); RACT (Reactant or reagent)
(reduction; mechanism of triphenylphosphine oxidation by sterically protected sulfenic and selenenic acids, oxygen isotope exchange with water)

RE.CNT 22 THERE ARE 22 CITED REFERENCES AVAILABLE FOR THIS RECORD

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L54 ANSWER 3 OF 12 HCAPLUS COPYRIGHT 2005 ACS on STN
AN 2002:269384 HCAPLUS
DN 136:401369
ED Entered STN: 11 Apr 2002
TI Mechanism of the Second Sulfenylation of Indole
AU Hamel, Pierre
CS The Merck Frosst Centre for Therapeutic Research, Pointe-Claire-Dorval, QC, H9R 4P8, Can.
SO Journal of Organic Chemistry (2002), 67(9), 2854-2858
CODEN: JOCEAH; ISSN: 0022-3263
PB American Chemical Society
DT Journal
LA English
CC 22-6 (Physical Organic Chemistry)
OS CASREACT 136:401369
AB Sulfenylation of indole using a sulfenyl chloride occurs initially at the 3-position of the ring, leading to a 3-indolyl sulfide. When an excess of sulfenyl chloride is used, a second sulfide group is introduced at the 2-position, and an indolyl 2,3-bis-sulfide results. We have demonstrated that this second sulfenylation occurs not by direct introduction of the second sulfide at the 2-position but via initial formation of an indolenium 3,3-bis-sulfide intermediate, followed by migration of one of the sulfide groups to the 2-position. This was achieved by the isolation of two examples of 3H-indole 3,3-bis-sulfides and by subsequent demonstration that they rearrange to the indolyl 2,3-bis-sulfides by treatment with sulfenyl halides.
ST indole sulfenylation mechanism
IT Rearrangement
(of 3H-indole 3,3-bis-sulfides; mechanism of the second sulfenylation of indole with sulfenyl chlorides)
IT **Sulfenyl compounds**
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); RCT (Reactant); **SPN (Synthetic preparation)**; **PREP (Preparation)**; PROC (Process); RACT (Reactant or reagent)
(sulfenyl **chlorides**; mechanism of the second sulfenylation of indole with sulfenyl **chlorides**)
IT **Substitution reaction**
(**sulfenylation**; mechanism of the second **sulfenylation** of indole with **sulfenyl** chlorides)
IT **Quaternization**
(using **sulfenyl** chloride; mechanism of the second **sulfenylation** of indole with **sulfenyl** chlorides)
IT 120-72-9, Indole, reactions 54491-43-9, 3-(Phenylthio)indole
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); RCT (Reactant); PROC (Process); RACT (Reactant or reagent)
(mechanism of the second sulfenylation of indole with sulfenyl chlorides)
IT **931-59-9P**, Benzenesulfenyl **chloride** 933-00-6P, p-Toluenesulfenyl chloride

RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); RCT (Reactant); **SPN (Synthetic preparation); PREP (Preparation)**; PROC (Process); RACT (Reactant or reagent)
(mechanism of the second sulfenylation of indole with sulfenyl **chlorides)**

IT 120517-31-9, 2-(Phenylthio)indole
RL: RCT (Reactant); RACT (Reactant or reagent)
(mechanism of the second sulfenylation of indole with sulfenyl chlorides)

IT 647024-85-9P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(mechanism of the second sulfenylation of indole with sulfenyl chlorides)

IT 70291-88-2P, 2,3-Bis(phenylthio)indole 200617-54-5P, 3-(Phenylthio)-3-(p-tolylthio)-3H-indole 431878-78-3P, 3-(Phenylthio)-2-(p-tolylthio)indole 431878-79-4P, 2-(Phenylthio)-3-(p-tolylthio)indole 431878-81-8P, 2,3,3-Tris(phenylthio)-3H-indole
RL: SPN (Synthetic preparation); PREP (Preparation)
(mechanism of the second sulfenylation of indole with sulfenyl chlorides)

IT 200617-53-4P, 3,3-Bis(phenylthio)-3H-indole
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); PROC (Process); RACT (Reactant or reagent)
(rearrangement; mechanism of the second sulfenylation of indole with sulfenyl chlorides)

IT 431878-80-7P, 2,3-Bis(p-tolylthio)indole
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(regioselective desulfenylation; mechanism of the second sulfenylation of indole with sulfenyl chlorides)

RE.CNT 14 THERE ARE 14 CITED REFERENCES AVAILABLE FOR THIS RECORD

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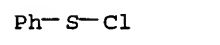
IT **931-59-9P**, Benzenesulfenyl **chloride**
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); RCT (Reactant); **SPN (Synthetic preparation); PREP (Preparation)**; PROC (Process); RACT (Reactant or reagent)
(mechanism of the second sulfenylation of indole with sulfenyl **chlorides)**

RN 931-59-9 HCAPLUS
CN Benzenesulfenyl chloride (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)



L54 ANSWER 4 OF 12 HCAPLUS COPYRIGHT 2005 ACS on STN
AN 2000:85193 HCAPLUS
DN 132:207478
ED Entered STN: 04 Feb 2000

TI A new sulfenylation reagent, 3-phenylsulfenyl-2-(N-cyanoimino)thiazolidine, and its optically active version
AU Tanaka, Tetsuaki; Azuma, Tsutomu; Fang, Xie; Uchida, Shuji; Iwata, Chuzo; Ishida, Toshimasa; In, Yasuko; Maezaki, Naoyoshi
CS Graduate School Pharmaceutical Sciences, Osaka Univ., Suita, 565, Japan
SO Synlett (2000), (1), 33-36
CODEN: SYNLES; ISSN: 0936-5214
PB Georg Thieme Verlag
DT Journal
LA English
CC 21-2 (General Organic Chemistry)
Section cross-reference(s): 75
OS CASREACT 132:207478
AB A new sulfenylation reagent, 3-(phenylthio)-2-(N-cyanoimino)thiazolidine (I), was developed, which is readily available and stable upon storage. Compound I easily reacts with amines or thiols to give the corresponding sulfenamides or asym. disulfides in excellent yields. The α-sulfenylation of carbonyl compds. with I proceeds smoothly. Furthermore, the optically active 4-diphenylmethyl derivative of I was synthesized as an asym. sulfenylation reagent, which realized 96% ee upon α-sulfenylation of a cyclic β-keto ester.
ST phenyl cyaniminothiazolidinyl sulfide sulfenylation reagent prepn; phenylsulfenylcyaniminothiazolidine stereoselective sulfenylation reagent; amine phenylsulfenylcyaniminothiazolidine sulfenylation; thiol phenylsulfenylcyaniminothiazolidine sulfenylation; sulfenamide prepn; disulfide prepn; ketone phenylsulfenylcyaniminothiazolidine sulfenylation
IT Thioethers
RL: SPN (Synthetic preparation); PREP (Preparation)
(oxo; sulfenylation with (phenylthio)(cyanimino)thiazolidine as reagent)
IT Amides, preparation
Amides, preparation
Sulfenyl compounds
Sulfenyl compounds
RL: SPN (Synthetic preparation); PREP (Preparation)
(sulfenamides; sulfenylation with (phenylthio)(cyanimino)thiazolidine as reagent)
IT Amines, reactions
Reagents
Thiols (organic), reactions
RL: **RCT (Reactant); RACT (Reactant or reagent)**
(sulfenylation with (phenylthio)(cyanimino)thiazolidine as reagent)
IT Ketones, preparation
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(sulfenylation with (phenylthio)(cyanimino)thiazolidine as reagent)
IT Disulfides
RL: SPN (Synthetic preparation); PREP (Preparation)
(sulfenylation with (phenylthio)(cyanimino)thiazolidine as reagent)
IT Addition reaction
Substitution reaction
(sulfenylation; sulfenylation with (phenylthio)(cyanimino)thiazolidine as reagent)
IT 258823-04-0P
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(crystal structure)
IT 35874-99-8P 76643-99-7P 84978-78-9P
RL: BYP (Byproduct); PREP (Preparation)
(disulfenylation byproducts from the preparation of monosulfenylated ketones with (phenylthio)(cyanimino)thiazolidine as reagent)
IT 868-59-7 10191-60-3
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation of (phenylthio)(cyanimino)thiazolidine as stereoselective sulfenylation reagent)
IT 258822-90-1P 258822-91-2P 258822-92-3P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT

(Reactant or reagent)
(preparation of (phenylthio)(cyanimino)thiazolidine as stereoselective sulfenylation reagent)

IT 258822-93-4P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(stereoselective sulfenylation reagent)

IT 258823-03-9P
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(stereoselective sulfenylation with (phenylthio)(cyanimino)thiazolidine)

IT 609-14-3, Ethyl α-methylacetoacetate 874-23-7, 2-Acetylcyclohexanone 1655-07-8, Ethyl 2-oxocyclohexanecarboxylate 3580-38-9, 2-Benzoylcyclohexanone 4513-77-3, 2-Cyanocyclohexanone 10472-24-9 41043-87-2, 2-Pivaloylcyclohexanone 41302-34-5, Methyl 2-oxocyclohexanecarboxylate 52784-32-4 98969-09-6 258822-94-5
RL: RCT (Reactant); RACT (Reactant or reagent)
(stereoselective sulfenylation with (phenylthio)(cyanimino)thiazolidine)

IT 65755-68-2P 72302-36-4P 98711-81-0P 258822-95-6P 258822-96-7P 258822-97-8P 258822-98-9P 258822-99-0P 258823-00-6P 258823-01-7P 258823-02-8P
RL: SPN (Synthetic preparation); PREP (Preparation)
(stereoselective sulfenylation with (phenylthio)(cyanimino)thiazolidine)

IT 258822-89-8
RL: RCT (Reactant); RACT (Reactant or reagent)
(sulfenylation reagent)

IT 62-53-3, Phenylamine, reactions 75-04-7, Ethylamine, reactions **75-08-1, Ethanethiol** 75-64-9, tert-Butylamine, reactions 100-46-9, Benzylamine, reactions 108-91-8, Cyclohexylamine, reactions 108-94-1, Cyclohexanone, reactions 110-89-4, Piperidine, reactions 120-92-3, Cyclopentanone 123-75-1, Pyrrolidine, reactions 141-97-9, Ethyl acetoacetate 502-42-1, Cycloheptanone 529-34-0, 1-Tetralone 583-60-8, 2-Methylcyclohexanone 26445-03-4
RL: **RCT (Reactant); RACT (Reactant or reagent)**
(sulfenylation with (phenylthio)(cyanimino)thiazolidine as reagent)

IT 4032-81-9P 14933-91-6P 19117-31-8P 20841-46-7P 23837-23-2P 24380-80-1P 27872-73-7P 27920-40-7P, 2-(Phenylthio)cyclohexanone 29959-86-2P 34780-06-8P 36476-32-1P 52190-40-6P, 2-(Phenylthio)cyclopentanone 52190-41-7P, 2-(Phenylthio)cycloheptanone 54376-46-4P, 2-Methyl-2-(phenylthio)cyclohexanone 79581-44-5P 95156-44-8P
RL: SPN (Synthetic preparation); PREP (Preparation)
(sulfenylation with (phenylthio)(cyanimino)thiazolidine as reagent)

RE.CNT 27 THERE ARE 27 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

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(8) Harpp, D; Tetrahedron Lett 1971, P4953 HCAPLUS
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(11) Iwata, C; Heterocycles 1990, V31, P1601 HCAPLUS
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(14) Iwata, C; Synthesis 1988, P261 HCAPLUS
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(16) Mukaiyama, T; Tetrahedron Lett 1970, P5115 HCAPLUS
(17) Murai, S; J Chem Soc, Chem Commun 1972, P946 HCAPLUS
(18) Neidlein, R; Arch Pharm 1972, V305, P731 HCAPLUS
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Search done by Ross Schipe

(20) Paterson, I; Synlett 1991, P145 HCAPLUS
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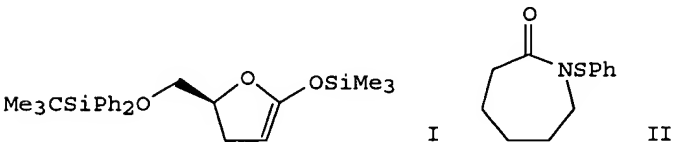
(22) Tanaka, T; Heterocycles 1996, V42, P265 HCAPLUS
(23) Tanaka, T; J Chem Soc, Chem Commun 1994, P2301 HCAPLUS
(24) Tanaka, T; Synlett 1997, P316 HCAPLUS
(25) Trost, B; Chem Rev 1978, V78, P363 HCAPLUS
(26) Trost, B; J Am Chem Soc 1976, V98, P4887 HCAPLUS
(27) Yura, T; Chem Lett 1986, P1809 HCAPLUS
IT **75-08-1, Ethanethiol**
RL: **RCT (Reactant); RACT (Reactant or reagent)**
(sulfenylation with (phenylthio)(cyanimino)thiazolidine as reagent)
RN 75-08-1 HCAPLUS
CN Ethanethiol (8CI, 9CI) (CA INDEX NAME)

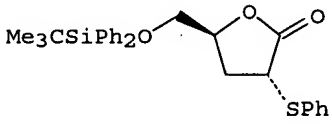
H_3C-CH_2-SH

L54 ANSWER 5 OF 12 HCAPLUS COPYRIGHT 2005 ACS on STN
AN 1992:151460 HCAPLUS
DN 116:151460
ED Entered STN: 17 Apr 1992
TI Diastereoselective sulfenylation reactions employing N-phenylthio lactams under nonbasic conditions
AU Wilson, Lawrence J.; Liotta, Dennis C.
CS Dep. Chem., Emory Univ., Atlanta, GA, 30322, USA
SO Journal of Organic Chemistry (1992), 57(7), 1948-50
CODEN: JOCEAH; ISSN: 0022-3263
DT Journal
LA English
CC 27-6 (Heterocyclic Compounds (One Hetero Atom))
OS CASREACT 116:151460
GI



I

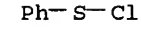
II



III

AB Silyl enol ethers and silyl ketene acetals react with sulfenamides in the presence of trimethylsilyl triflate to give the corresponding trans-sulfenylated ketones and lactones. The stereoselectivity observed in these reactions is good to excellent and appears to be governed by steric factors. Thus, the silyl ketene acetal I reacted with N-phenylthio-ε-caprolactam II in presence of trimethylsilyl triflate to give 82% trans-sulfenylated lactone III and 10% of cis-sulfenylated lactone.
ST lactone sulfenylation **phenylthiolactam** stereoselectivity; ketone sulfenylation **phenylthiolactam** stereoselectivity
IT Stereochemistry
(of sulfenylation of ketones and lactones via silyl enol ethers and silyl ketene acetals and **phenylthiolactams**)

IT **Substitution reaction**
(**sulfenylation**, stereoselective, of ketones and lactones via silyl enol ethers and silyl ketene acetals with **phenylthiolactams**)
IT 125440-13-3
RL: **RCT (Reactant)**; **RACT (Reactant or reagent)**
(diastereoselective sulfenylation of, with **phenylthiolactams**)
IT 36452-23-0P 139524-50-8P 139524-51-9P 139524-52-0P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and diastereoselective sulfenylation by, of silyl enol ethers and silyl ketene acetals)
IT **931-59-9P**, Benzenesulfenyl **chloride**
RL: **SPN (Synthetic preparation)**; **PREP (Preparation)**
(preparation and sulfenylation by, of dimethylpiperidinone)
IT 52190-40-6P 62675-41-6P 62675-50-7P 63608-45-7P 66032-98-2P
66032-99-3P 73843-08-0P 129778-50-3P 129778-55-8P 139524-53-1P
139524-54-2P 139524-55-3P 139563-96-5P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
IT 98-53-3 583-60-8 130745-59-4
RL: **RCT (Reactant)**; **RACT (Reactant or reagent)**
(sequential conversion to silyl enol ether and sulfenylation of, with **phenylthiolactam**)
IT 108-29-2
RL: **RCT (Reactant)**; **RACT (Reactant or reagent)**
(sequential conversion to silyl ketene acetal and diastereoselective sulfenylation of, with **phenylthiolactam**)
IT 102717-29-3
RL: **RCT (Reactant)**; **RACT (Reactant or reagent)**
(sequential conversion to silyl ketene acetal and diastereoselective sulfenylation of, with **phenylthiolactams**)
IT 19980-43-9
RL: RCT (Reactant); RACT (Reactant or reagent)
(sulfenylation of)
IT 139524-56-4
RL: RCT (Reactant); RACT (Reactant or reagent)
(N-sulfenylation of, with phenylsulfenyl hloride)
IT **931-59-9P**, Benzenesulfenyl **chloride**
RL: **SPN (Synthetic preparation)**; **PREP (Preparation)**
(preparation and sulfenylation by, of dimethylpiperidinone)
RN 931-59-9 HCAPLUS
CN Benzenesulfenyl chloride (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)



L54 ANSWER 6 OF 12 HCAPLUS COPYRIGHT 2005 ACS on STN
AN 1991:121017 HCAPLUS
DN 114:121017
ED Entered STN: 06 Apr 1991
TI Mechanistic aspects of nucleophilic substitutions of sulfenic acid derivatives
AU Okuyama, Tadashi
CS Fac. Eng. Sci., Osaka Univ., Toyonaka, 560, Japan
SO Chem. Sulphenic Acids Their Deriv. (1990), 743-63. Editor(s): Patai, Saul. Publisher: Wiley, Chichester, UK.
CODEN: 57ARAT
DT Conference; General Review
LA English
CC 22-0 (Physical Organic Chemistry)
AB Sulfenium ion intermediates; bimol. nucleophilic substitution of sulfenyl halides, sulfenate esters, and disulfides; 70 refs.
ST review sulfenic acid nucleophile substitution
IT **Sulfenic acids**

RL: RCT (Reactant); RACT (Reactant or reagent)
(derivs., nucleophilic substitution of, mechanism of)

IT Disulfides
Sulfenyl halides
RL: RCT (Reactant); RACT (Reactant or reagent)
(nucleophilic substitution of, mechanism of)

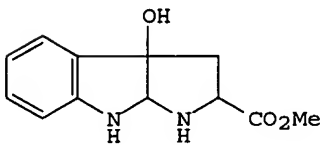
IT **Substitution reaction, nucleophilic**
(of sulfenic acid derivs., mechanism of)

IT **Thiols, reactions**
RL: **RCT (Reactant); RACT (Reactant or reagent)**
(sulfonate and sulfinate derivs., nucleophilic substitution of, mechanism of)

IT **Sulfenic acids**
RL: RCT (Reactant); RACT (Reactant or reagent)
(esters, nucleophilic substitution of, mechanism of)

IT 18155-21-0D, Sulfonium, derivs.
RL: PROC (Process)

L54 ANSWER 7 OF 12 HCAPLUS COPYRIGHT 2005 ACS on STN
AN 1987:497110 HCAPLUS
DN 107:97110
ED Entered STN: 19 Sep 1987
TI Analgesic dipeptide derivatives. 3. Synthesis and structure-activity relationships of o-nitrophenyl-modified analogs of the analgesic compound H-Lys-Trp(NPS)-OMe
AU Garcia-Lopez, M. Teresa; Gonzalez-Muniz, Rosario; Molinero, M. Teresa; Naranjo, Jose R.; Del Rio, J.
CS Inst. Quim. Med., CSIC, Madrid, 28006, Spain
SO Journal of Medicinal Chemistry (1987), 30(9), 1658-63
CODEN: JMCMAR; ISSN: 0022-2623
DT Journal
LA English
CC 34-3 (Amino Acids, Peptides, and Proteins)
Section cross-reference(s): 1
OS CASREACT 107:97110
GI



II

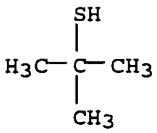
AB Title dipeptides H-Lys-Trp(SR)-OMe [I; R = Ph, C6H4CO2Me-o, C6H4NO2-p, C6H3(NO2)-2,4] were prepared by sulfenylating Z-Lys(Z)-Trp-OMe (Z = PhCH2O2C) with RSCl and Z-deblocking the resulting Z-Lys(Z)-Trp(SR)-OMe by (CF3CO2)3B/CF3CO2H or Me3SiI/MeCN. I (R = C6H4NHAc-o) was prepared from Z-Lys(Z)-Trp(SC6H4NO2-o)-OMe by sequential reduction, N-acetylation, and Z-deblocking. Pyrrolol[2,3-b]indole II was treated with Me3CSH and HSCH2CH2CO2Me to give H-Trp(SCMe3)-OMe and H-Trp(SCH2CH2CO2Me)-OMe, resp., which were used in the synthesis of I (R = CMe3 and CH2CH2CO2Me, resp.). I (R = C6H4CO2Me-o) elicited a naloxone-reversible antinociceptive effect in mice similar to that of the analgesic dipeptide I (R = C6H4NO2-o). No analgesia was observed for the other I. Structure-activity studies indicated that the role of the SC6H4NO2-o and SC6H4CO2Me-o moieties could be related to the adoption of a preferential active conformation.

ST phenylsulfenyl tryptophan dipeptide prepn analgesia; sulfenylation tryptophan dipeptide; structure analgesic phenylsulfenyl tryptophan dipeptide

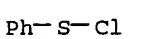
IT Analgesics

(lysyl-(nitrophenylsulfenyl)tryptophan Me ester analogs)
IT Molecular structure-biological activity relationship
(analgesic, of lysyl-(nitrophenylsulfenyl)tryptophan Me ester analogs)
IT **Substitution reaction**
(**sulfenylation**, of tryptophan-containing dipeptide)
IT 109064-70-2
RL: BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); BIOL (Biological study)
(analgesic activity of)
IT 5459-63-2
RL: RCT (Reactant); RACT (Reactant or reagent)
(disulfide cleavage-S-chlorination of)
IT 6066-82-6, N-Hydroxysuccinimide
RL: RCT (Reactant); RACT (Reactant or reagent)
(esterification by, of lysine derivative)
IT 405-39-0
RL: RCT (Reactant); RACT (Reactant or reagent)
(esterification of, with hydroxysuccinimide)
IT 109064-77-9P 109064-78-0P 109064-79-1P 109064-80-4P 109064-81-5P 109064-85-9P
RL: BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation)
(preparation and analgesic activity of)
IT 109064-72-4P 109064-73-5P 109064-74-6P 109064-75-7P 109064-76-8P 109064-84-8P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and deblocking of)
IT 109064-86-0P 109064-87-1P 109064-88-2P 109064-89-3P 109064-90-6P 109064-91-7P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and peptide coupling of, with lysine derivative)
IT 21160-83-8P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and peptide coupling of, with tryptophan derivs.)
IT 109064-82-6P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and reduction of)
IT 78880-71-4P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and sulfenylation by, of tryptophan-containing dipeptide)
IT 109064-83-7P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and N-acetylation of)
IT 7652-46-2
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with tryptophan-containing dipeptide)
IT **75-66-1**, tert-**Butylthiol** 2935-90-2, 3-Mercaptopropionic acid methyl ester
RL: **RCT (Reactant)**; **RACT (Reactant or reagent)**
(ring cleavage by, of hexahydropyrroloindole derivative)
IT 51440-62-1
RL: **RCT (Reactant)**; **RACT (Reactant or reagent)**
(ring cleavage of, by **thiols**)
IT 528-76-7 931-59-9 937-32-6 7669-54-7
RL: RCT (Reactant); RACT (Reactant or reagent)
(sulfenylation by, of tryptophan-containing dipeptide)
IT 52126-94-0
RL: RCT (Reactant); RACT (Reactant or reagent)
(sulfenylation of)
IT **75-66-1**, tert-**Butylthiol**

RL: RCT (Reactant); RACT (Reactant or reagent)
(ring cleavage by, of hexahydropyrroloindole derivative)
RN 75-66-1 HCAPLUS
CN 2-Propanethiol, 2-methyl- (8CI, 9CI) (CA INDEX NAME)



IT 931-59-9
RL: RCT (Reactant); RACT (Reactant or reagent)
(sulfenylation by, of tryptophan-containing dipeptide)
RN 931-59-9 HCAPLUS
CN Benzenesulfenyl chloride (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)



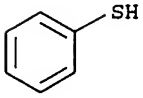
L54 ANSWER 8 OF 12 HCAPLUS COPYRIGHT 2005 ACS on STN
AN 1981:514988 HCAPLUS
DN 95:114988
ED Entered STN: 12 May 1984
TI Participation of electrophilic organic compounds of sulfur(II) in catalytic conversions. II. Catalytic exchange reaction of N-monosulfenated amines with S-esters of thiocarboxylic acids
AU Parfenov, E. A.; Fomin, V. A.
CS Vses. Nauchno-Issled. Vitam. Inst., Moscow, USSR
SO Zhurnal Obshchei Khimii (1981), 51(5), 1129-37
CODEN: ZOKHA4; ISSN: 0044-460X
DT Journal
LA Russian
CC 25-18 (Noncondensed Aromatic Compounds)
Section cross-reference(s): 6
AB RSX (R = Ph, 2-O2NC6H4; X = Cl, Br) reacted with R1R2NH (R1R2N = piperidino, morpholino, PhNH, PhNMe, α-C10H7NH, carbazolyl, benzimidazolyl) in the presence of Et3N to give 8 corresponding RSNR1R2 (I) in 32-88% yield. I reacted with 6 R3COSR4 (R3 = Me, Ph, CH2:CHCH2CH2, 3-pyridyl; R4 = CH2Ph, Ph, C6H4NO2-2 and -4) in the presence of a soft base [Ph3P, P(OMe)3, P(OPh)3] to give 9 corresponding R3CONR1R2 and RSSR4 in ≥81% yield. This is a model reaction for energy transfer in living systems.
ST energy transfer model living system; exchange reaction sulfenamide thiocarboxylate
IT Energy transfer
(in living systems, exchange reaction of sulfenamides with **thiolcarboxylates** as model for)
IT **Exchange reaction**
(of **sulfenamides** with **thiolcarboxylates**, as model for energy transfer in living systems)
IT **Sulfenamides**
RL: **RCT (Reactant)**; SPN (Synthetic preparation); PREP (Preparation); **RACT (Reactant or reagent)**
(preparation and exchange reaction of, with **thiolcarboxylates**)
IT Amines, reactions
RL: RCT (Reactant); RACT (Reactant or reagent)
(sulfenylation of)
IT Exchange reaction catalysts
(trivalent phosphorus compds., for sulfenamides with **thiolcarboxylates**)
IT Esters, reactions

RL: RCT (Reactant); RACT (Reactant or reagent)
(thiol, exchange reaction of, with sulfenamides)
IT 98-88-4
RL: RCT (Reactant); RACT (Reactant or reagent)
(acylation of nitrothiophenol with)
IT 112-67-4
RL: RCT (Reactant); RACT (Reactant or reagent)
(acylation of toluenethiol with)
IT 100-53-8
RL: RCT (Reactant); RACT (Reactant or reagent)
(acylation of, with palmitoyl chloride)
IT 4875-10-9
RL: RCT (Reactant); RACT (Reactant or reagent)
(benzoylation of)
IT 101-02-0 121-45-9 603-35-0, uses and miscellaneous
RL: CAT (Catalyst use); USES (Uses)
(catalysts, for exchange reaction of sulfenamides with thiolcarboxylates)
IT 1155-00-6
RL: RCT (Reactant); RACT (Reactant or reagent)
(esterification of pyridinecarboxylic acid with)
IT 59-67-6, reactions
RL: RCT (Reactant); RACT (Reactant or reagent)
(esterification of, with bis(nitrophenyl) disulfide)
IT 591-80-0
RL: RCT (Reactant); RACT (Reactant or reagent)
(esterification of, with di-Ph disulfide)
IT 884-09-3P 934-87-2P 15119-62-7P 24197-72-6P 78945-05-8P
78966-71-9P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and exchange reaction of, with sulfenamides)
IT 4837-33-6P 7257-62-7P 14933-91-6P 33224-04-3P 66552-58-7P
78945-06-9P 78945-08-1P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and exchange reaction of, with thiolcarboxylates)
IT 93-98-1P 103-84-4P 108-98-5P, preparation 492-85-3P
618-42-8P 634-42-4P 776-75-0P 1934-92-5P 58170-51-7P 66591-30-8P
78945-04-7P 78945-07-0P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
IT 882-33-7
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with pentenoic acid)
IT 98-74-8
RL: RCT (Reactant); RACT (Reactant or reagent)
(reduction of, in presence of acetic acid, thioacetate by)
IT 98-09-9
RL: RCT (Reactant); RACT (Reactant or reagent)
(reduction of, with red phosphorus in presence of acetic and benzoic acids, thiolcarboxylates by)
IT 22024-99-3 28074-23-9
RL: RCT (Reactant); RACT (Reactant or reagent)
(sulfenylation of amines with)
IT 7669-54-7
RL: RCT (Reactant); RACT (Reactant or reagent)
(sulfenylation of carbazole with)
IT 62-53-3, reactions
RL: RCT (Reactant); RACT (Reactant or reagent)
(sulfenylation of, with benzene- and nitrobenzenesulfenyl bromide)
IT 51-17-2 100-61-8, reactions 110-89-4, reactions 110-91-8, reactions
134-32-7
RL: RCT (Reactant); RACT (Reactant or reagent)
(sulfenylation of, with nitrobenzenesulfenyl bromide)
IT 86-74-8

RL: RCT (Reactant); RACT (Reactant or reagent)
(sulfenylation of, with nitrobenzenesulfenyl chloride)
IT **100-53-8**
RL: **RCT (Reactant); RACT (Reactant or reagent)**
(acylation of, with palmitoyl chloride)
RN 100-53-8 HCAPLUS
CN Benzenemethanethiol (9CI) (CA INDEX NAME)

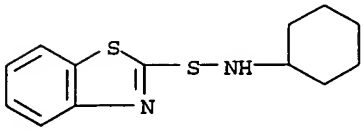
HS—CH_2—Ph

IT **108-98-5P**, preparation
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 108-98-5 HCAPLUS
CN Benzenethiol (8CI, 9CI) (CA INDEX NAME)

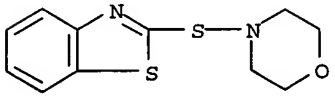


L54 ANSWER 9 OF 12 HCAPLUS COPYRIGHT 2005 ACS on STN
AN 1981:65055 HCAPLUS
DN 94:65055
ED Entered STN: 12 May 1984
TI Reaction of sulfenamides with dialkyl and trialkyl phosphites. An efficient synthesis of phosphoramidates by unusual substitution at the sulfur-nitrogen bond in (2-benzothiazolyl)sulfenamides
AU Torii, Sigeru; Sayo, Noboru; Tanaka, Hideo
CS Sch. Eng., Okayama Univ., Okayama, 700, Japan
SO Chemistry Letters (1980), (6), 695-8
CODEN: CMLTAG; ISSN: 0366-7022
DT Journal
LA English
CC 23-12 (Aliphatic Compounds)
Section cross-reference(s): 22
OS CASREACT 94:65055
AB Regioselective attack of the trivalent P atom of dialkyl and trialkyl phosphites on either N or S atom of RSNR1R2 (R = Ph, 2-benzothiazolyl; R1, R2 = H, alkyl, NR1R2 = succinimidyl, morpholino) has been found. The reaction of phenylsulfenamides with dialkyl phosphites yielded **phosphorothiolates**, whereas the treatment of (2-benzothiazolyl)sulfenamides with dialkyl and trialkyl phosphites gave phosphoramidates in excellent yields.
ST sulfenamide reaction phosphite regioselectivity
IT **Substitution reaction**
(in reaction of **sulfenamides** with dialkyl and trialkyl phosphites, regioselectivity in)
IT Stereochemistry
(regioselectivity, in reaction of sulfenamides with dialkyl and trialkyl phosphites)
IT 597-24-0P 597-25-1P 1889-58-3P 2672-32-4P 3167-69-9P 4237-00-7P
4972-36-5P 15267-38-6P 20465-00-3P 22685-19-4P 22685-20-7P
32405-88-2P 37097-43-1P 53640-96-3P 54480-52-3P 59658-74-1P
67828-17-5P 68036-32-8P 74124-44-0P 74124-45-1P 74124-46-2P
74124-48-4P 74124-50-8P 74124-51-9P 74130-08-8P 75291-13-3P
75291-14-4P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
IT 95-31-8 95-33-0 102-77-2 2720-65-2 4837-31-4
10220-34-5 14204-24-1 26773-65-9 26773-69-3 63451-39-8

66552-53-2
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with phosphites)
IT 691-96-3 762-04-9 868-85-9
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with sulfenamides)
IT **95-33-0 102-77-2**
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with phosphites)
RN 95-33-0 HCAPLUS
CN 2-Benzothiazolesulfenamide, N-cyclohexyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)



RN 102-77-2 HCAPLUS
CN Morpholine, 4-(2-benzothiazolylthio)- (9CI) (CA INDEX NAME)



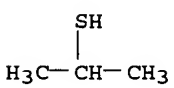
L54 ANSWER 10 OF 12 HCAPLUS COPYRIGHT 2005 ACS on STN
AN 1979:22526 HCAPLUS
DN 90:22526
ED Entered STN: 12 May 1984
TI Action of sulfenyl chlorides on N-alkoxyureas
AU Demoute, Jean Pierre; Teche, Andre; Perronnet, Jacques
CS Cent. Rech. Roussel Uclaf, Romainville, Fr.
SO Journal of Chemical Research, Synopses (1978), (7), 244-5
CODEN: JRPSDC; ISSN: 0308-2342
DT Journal
LA English/French
CC 25-21 (Noncondensed Aromatic Compounds)
Section cross-reference(s): 23
OS CASREACT 90:22526
AB 3,4-Cl2C6H3NHCONRR1 (I, R = H, R1 = MeO) reacted with ClSR2 (II, R2 = Et, Me2CH, Ph, p-O2NC6H4) to give 22-75% I (R = H, R1 = SR2). Similar **alkylthiolation** was observed in the reactions of I (R = H, R1 = PhCH2O, EtO) with ClSEt, and of I (R = H, R1 = PhCH2O) with ClSCCl3. I (R = Me2CHS, PhS, R1 = Me3CO; R = Cl3CS, R1 = MeO) were obtained in 52-80% yield by the reaction of II (R2 = Me2CH, Ph, Cl3C) with I (R = H; R = Me3CO, MeO). I (R = Cl3CS, R1 = MeO) underwent an elimination reaction on thermolysis to give I (R = Cl3CS, R1 = H). The Na salts of I (R = H; R1 = EtS, PhS) underwent further **alkylthiolation** with II (R2 = Et, Ph) to give 23-67% (R = R1 = EtS, PhS; R = EtS, R1 = PhS). Similarly, O-methyl-N-acylhydroxylamines were converted to the resp. N-(isopropylthio)-N-acylhydroxylamines.
ST alkanesulfenyl chloride substitution alkoxyphenylurea; phenylurea alkoxy dealkoxylation **alkylthiolation**; dealkoxylation **alkylthiolation** alkoxyphenylurea; methoxyamine demethoxylation **alkylthiolation**; urea phenyl alkylthio; benzenesulfenyl chloride substitution alkoxyphenylurea
IT **Sulfenyl chlorides**
RL: **RCT (Reactant); RACT (Reactant or reagent)**
(dealkoxylation-**alkylthiolation** of alkoxyureas by)

IT Substitution reaction
(dealkoxylation-alkylthiolation, of alkoxyphenylureas by sulfenyl chlorides)
IT 1496-75-9
RL: RCT (Reactant); RACT (Reactant or reagent)
(dealkoxylation-alkylthiolation of 1-alkoxy-3-phenylureas by)
IT 68692-28-4P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and dealkoxylation-alkylthiolation of)
IT 68692-34-2P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and demethoxylation of)
IT 60916-96-3P 68692-38-6P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and demethoxylation-alkylthiolation of)
IT 68692-39-7P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and demethoxylation-benzenethiolation of)
IT 68692-29-5P 68692-31-9P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and thioalkylation of)
IT 68692-30-8P 68692-32-0P 68692-33-1P 68692-35-3P 68692-36-4P
68692-37-5P 68692-40-0P 68692-41-1P 68692-42-2P 68692-43-3P
68692-44-4P 68692-45-5P 68692-46-6P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
IT 594-42-3 931-59-9 937-32-6 19760-04-4
RL: RCT (Reactant); RACT (Reactant or reagent)
(thioalkylation by, of alkoxyphenylureas)
IT 17356-61-5 51458-03-8
RL: RCT (Reactant); RACT (Reactant or reagent)
(thioalkylation of, with sulfenyl chlorides)
IT 52420-54-9
RL: RCT (Reactant); RACT (Reactant or reagent)
(thioethylation of, with ethylsulfenyl chloride)
IT 40750-59-2
RL: RCT (Reactant); RACT (Reactant or reagent)
(N-acylation by N-methoxycarbamate ester)
IT 68692-38-6
RL: RCT (Reactant); RACT (Reactant or reagent)
(N-acylation of aniline derivative by)
IT 79-44-7 7693-46-1
RL: RCT (Reactant); RACT (Reactant or reagent)
(N-acylation of methoxyamine by)
IT 67-62-9
RL: RCT (Reactant); RACT (Reactant or reagent)
(N-acylation reactions with chloroformate ester and carbamoyl chloride derivative)
IT 31225-17-9
RL: PROC (Process)
(O-tert-butoxylation of)
IT 931-59-9
RL: RCT (Reactant); RACT (Reactant or reagent)
(thioalkylation by, of alkoxyphenylureas)
RN 931-59-9 HCAPLUS
CN Benzenesulfenyl chloride (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

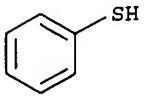
Ph—S—Cl

L54 ANSWER 11 OF 12 HCAPLUS COPYRIGHT 2005 ACS on STN
AN 1974:551895 HCAPLUS
DN 81:151895

ED Entered STN: 12 May 1984
TI Novel sulfenylation reagent. Synthesis and reactions of N-(arylthio)isatins
AU Furukawa, Mitsuru; Suda, Tchiaki; Hayashi, Seigoro
CS Fac. Pharm. Sci., Kumamoto Univ., Kumamoto, Japan
SO Chemistry Letters (1974), (8), 881-2
CODEN: CMLTAG; ISSN: 0366-7022
DT Journal
LA English
CC 27-11 (Heterocyclic Compounds (One Hetero Atom))
OS CASREACT 81:151895
GI For diagram(s), see printed CA Issue.
AB N-(Arylthio)isatins (I, R = Ph, 3-MeC6H4, PhCH2, 2-O2NC6H4, 4-ClC6H4) were prepared in 73-83% yields by treatment of isatin with RSCl. I were efficient sulfur-transfer reagents and react with organometallic compds., thiols and NaCN to give sulfides, disulfides and thiocyanates.
ST isatin arylthio; sulfenyl chloride isatin reaction; sulfide aryl; disulfide aryl; thiocyanate aryl
IT **Sulfenylation**
(reagents, N-(arylthio)isatins as)
IT 74-96-4 **75-33-2** 108-40-7 108-86-1 **108-98-5** 109-72-8, reactions 591-51-5
RL: **RCT (Reactant)**; **RACT (Reactant or reagent)**
(Grignard reaction with N-(arylthio)isatins)
IT 139-66-2P 622-38-8P 770-34-3P 882-33-7P 2769-30-4P 3012-37-1P 5184-47-4P 5285-87-0P 6263-62-3P 13865-48-0P 35379-08-9P 53888-02-1P 53888-03-2P 53888-04-3P 53888-05-4P 53888-06-5P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
IT 143-33-9
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with N-(arylthio)isatins)
IT 91-56-5
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with arylsulfenyl chlorides)
IT **931-59-9** 933-01-7 7669-54-7 26826-81-3 38364-78-2
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with isatin)
IT **75-33-2** **108-98-5**
RL: **RCT (Reactant)**; **RACT (Reactant or reagent)**
(Grignard reaction with N-(arylthio)isatins)
RN 75-33-2 HCAPLUS
CN 2-Propanethiol (8CI, 9CI) (CA INDEX NAME)



RN 108-98-5 HCAPLUS
CN Benzenethiol (8CI, 9CI) (CA INDEX NAME)



IT **931-59-9**
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with isatin)
RN 931-59-9 HCAPLUS
CN Benzenesulfenyl chloride (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph—S—Cl

L54 ANSWER 12 OF 12 HCAPLUS COPYRIGHT 2005 ACS on STN
AN 1974:47395 HCAPLUS
DN 80:47395
ED Entered STN: 12 May 1984
TI Derivatives of β-chlorosulfenylcarboxylic acids and their reactions with nucleophilic reagents
AU Vasil'eva, T. P.; Lin'kova, M. G.; Kil'disheva, O. V.; Knunyants, I. L.
CS Inst. Elementoorg. Soedin., Moscow, USSR
SO Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya (1973), (10), 2379-82
CODEN: IASKA6; ISSN: 0002-3353
DT Journal
LA Russian
CC 23-12 (Aliphatic Compounds)
AB AcSH and six ClSCHR1CR2R3COX (R1 = H; R2 = H, Me; R3 = H, Cl; X = OMe, OH, Cl) form at 0° good yields of AcS2CHR1CR2R3COX. A similar reaction with liquid H2S gave four S(SCHR1CHR2COX)2 (R1 = H, Cl; R2 = H, Cl; X = OMe, OH) and the descending order of reactivity with H2S was: ClSCH2CH2CO2Me, ClSCH2CHClCO2Me, ClSCH2CHClCO2H, ClSCHClCH2CO2Me; ClSCHClCH2CO2H failed to react with H2S. The trisulfides decomposed during attempted distillation and formed S and corresponding disulfides. PhMgBr and ClSCH2CHRCO2Me readily gave PhSCH2CHRCO2Me (R = H, Cl).
ST aliph sulfenyl chloride substitution; hydrogen sulfide substitution reaction; nucleophilic substitution thiolacetic acid
IT **Sulfenyl chlorides**
RL: RCT (Reactant); RACT (Reactant or reagent)
(aliphatic, substitution reaction of, with nucleophiles)
IT Nucleophiles
(hydrogen sulfide and thiolacetic acid, substitution of, with aliphatic sulfenyl chlorides)
IT **Substitution reaction**
(of aliphatic sulfenyl chlorides with nucleophiles)
IT 108-86-1
RL: RCT (Reactant); RACT (Reactant or reagent)
(Grignard reaction of, with aliphatic sulfenyl chlorides)
IT 20707-94-2P 22198-59-0P 30826-41-6P 32371-96-3P 34036-98-1P
42801-75-2P 50962-23-7P 50962-24-8P 50962-25-9P 50962-26-0P
50962-27-1P 50962-28-2P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
IT 507-09-5, reactions 7783-06-4, reactions
RL: RCT (Reactant); RACT (Reactant or reagent)
(substitution reaction of, with aliphatic sulfenyl chlorides)
IT 41345-79-3
RL: RCT (Reactant); RACT (Reactant or reagent)
(substitution reaction of, with hydrogen sulfide)
IT 41345-74-8 41345-81-7 41345-82-8
RL: RCT (Reactant); RACT (Reactant or reagent)
(substitution reaction of, with nucleophiles)
IT 14274-18-1 14274-19-2 41345-80-6
RL: RCT (Reactant); RACT (Reactant or reagent)
(substitution reaction of, with thiolacetic acid)

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26, 1996), unless otherwise indicated in the original publications.

FILE CONTENT:1840 - 21 Aug 2005 VOL 143 ISS 8

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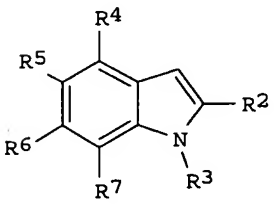
This file contains CAS Registry Numbers for easy and accurate substance identification.

=> d bib abs crd l78 tot

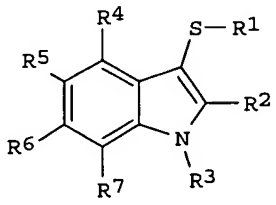
L78 ANSWER 1 OF 3 CASREACT COPYRIGHT 2005 ACS on STN
AN 141:106369 CASREACT
TI Method for 3-sulfenylation of indole-2-carboxylates by chlorination of thiols to sulfenyl chlorides
IN Wall Hamilton, Harriet; Krasutsky, Alexei P.; Reed, Jessica; Schlosser, Kevin
PA USA
SO U.S. Pat. Appl. Publ., 13 pp.
CODEN: USXXCO
DT Patent
LA English
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004133014	A1	20040708	US 2003-631268	20030731

PRAI US 2002-400092P 20020731
OS MARPAT 141:106369
GI



I



II

AB A highly efficient one-pot procedure for 3-sulfenylation of indole 2-carboxylates is described. Treatment of thiols of formula HSR1 [R1 = C1-6 alkyl, C2-6 alkoxycarbonyl, C3-7 cycloalkyl, C3-7 heterocycloalkyl, C1-6 alkyl-S(O)mRa, -C1-6 alkyl-S(O)mNRbRc, C1-6 alkyl-NRbRc, or C1-6 alkyl-CO-NRbRc, aryl, heteroaryl, etc.; m = 1, 2; Ra, Rb, Rc = H, C1-6 alkyl, C3-7 cycloalkyl, C3-6 heterocycloalkyl, or aryl] with N-chlorosuccinimide at -78° in CH2Cl2 affords sulfenyl chlorides of formula R1SCl in situ that readily react with indole 2-carboxylates [I; R2 = CO2H, tetrazolyl, C2-6 alkoxycarbonyl, CONRbRc (Rb, Rc = H, C1-6 alkyl), S(O)mRa, or -S(O)mNRbRc, NRbRc, etc. (wherein m = 1, 2; Ra, Rb, Rc = H, C1-6 alkyl, C3-7 cycloalkyl, C3-6 heterocycloalkyl, or aryl); R3 = H, each optionally substituted alkyl C1-6 or C1-6 alkanoyl; R4-R7 = H, halo, C1-6 alkyl, C1-6 alkoxy, cyano, C3-7

cycloalkyl, C3-7 heterocycloalkyl, C1-6 alkyl-S(O)mRa, -C1-6 -S(O)mNRbRc, C1-6 alkyl-NRbRc, or C1-6 alkyl-CO-NRbRc, C1-6 alkyl-COR1, S(O)mRa, S(O)mNRbRc, NRbRc, CONRbRc, aryl, heteroaryl, etc. (wherein m = 1, 2; Ra, Rb, Rc = H, C1-6 alkyl, C3-7 cycloalkyl, C3-6 heterocycloalkyl, heteroaryl or aryl)] to give 3-thioindoles (II) in high yields. This new method is milder, produces less waste, and is compatible with a wide range of **thiol** and indole functionality. It is also used for intramol. **sulfenylation** of N-(2-mercaptoethyl)-1H-indolecarboxamide derivs. to form a indole-fused 1,4-thiaazepane ring. Thus, to a cooled solution of N-chlorosuccinimide (2.74 g, 20.6 mmol) in CH2Cl2 (125 mL) at -78°, 3-methoxythiophenol (2.55 mL, 20.6 mmol) was added. The reaction was warmed to 0° over 15 min and a solution of indole-2-carboxylic acid Me ester (3 g, 17.1 mmol) in CH2Cl2 (25 mL) was added. The reaction stirred at 0° for 1 h, then concentrated under reduced pressure and the residue was suspended in H2O and stirred for 30 min to give, after filtration of the solid and recrystn. from EtOAc/hexanes to give 3-[(3-methoxyphenyl)thio]-1H-indole-2-carboxylic acid Me ester (3.22 g, 60%).
NO HIGHLIGHTING INFORMATION PRESENT

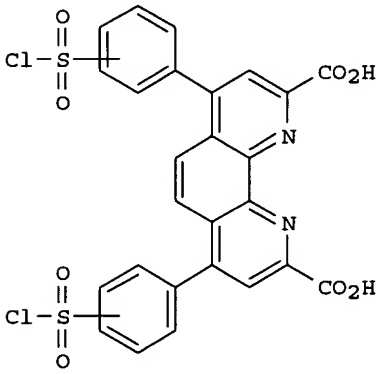
L78 ANSWER 2 OF 3 CASREACT COPYRIGHT 2005 ACS on STN
AN **140:253400** CASREACT
TI A highly efficient procedure for 3-**sulfenylation** of indole-2-carboxylates
AU **Schlosser, Kevin M.**; Krasutsky, Alexei P.; **Hamilton, Harriet W.**; **Reed, Jessica E.**; Sexton, Karen
CS Department of Chemistry, Pfizer Global Research and Development, Ann Arbor, MI, 48105, USA
SO Organic Letters (2004), 6(5), 819-821
CODEN: ORLEF7; ISSN: 1523-7060
PB American Chemical Society
DT Journal
LA English
AB A highly efficient one-pot procedure for 3-**sulfenylation** of 2-carboxyindoles is described. Treatment of **thiols** with N-chlorosuccinimide at -78 °C in CH2Cl2 affords **sulfenyl** chlorides in situ that readily react with 2-carboxyindoles to give 3-thioindoles in high yields. This new method is milder, produces less waste, and is compatible with a wide range of **thiol** and indole functionality.
NO HIGHLIGHTING INFORMATION PRESENT
RE.CNT 18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L78 ANSWER 3 OF 3 CASREACT COPYRIGHT 2005 ACS on STN
AN 128:47859 CASREACT
TI A ring opening reaction of benzisothiazolones. a new route to unsymmetrical disulfides
AU Sanchez, Joseph P.
CS Parke-Davis Pharmaceutical Research, Division of **Warner-Lambert** Company, Ann Arbor, MI, 48105, USA
SO Journal of Heterocyclic Chemistry (1997), 34(5), 1463-1467
CODEN: JHTCAD; ISSN: 0022-152X
PB HeteroCorporation
DT Journal
LA English
AB A series of unsym. disulfides has been prepared by employing a reaction involving a ring opening, nucleophilic attack of a **thiol** on a 1,2-benzisothiazol-3-one. The benzisothiazolones were in turn prepared by an intramol. ring closure of an amide on a **sulfenyl** thiocarbonate. The **sulfenyl** esters were synthesized as intermediates for preparing mixed-disulfides, but the benzisothiazolone ring closure occurred spontaneously. It was initially thought that the mixed-disulfides were being formed from the **sulfenyl** ester, but the isolation and stepwise reaction of the benzisothiazolones provided

proof for the reaction mechanism.
NO HIGHLIGHTING INFORMATION PRESENT
RE.CNT 25 THERE ARE 25 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

=> d bib abs crd l77 tot

L77 ANSWER 1 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN 138:205029 CASREACT
TI Synthesis and characterization of time resolved fluorescence immunoassay chelate reagent 4,7-bis(chlorosulfophenyl)-1,10-phenanthroline-2,9-dicarboxylic acid (BCPDA). (II)
AU Pan, Li-hua; Gao, Min; Lin, Min; Ding, Chun-li; Sun, Wen-wei; Zhao, Jie
CS Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun, 130022, Peop. Rep. China
SO Guangpuxue Yu Guangpu Fenxi (2002), 22(4), 573-576
CODEN: GYGFED; ISSN: 1000-0593
PB Beijing Daxue Chubanshe
DT Journal
LA Chinese
GI



I

AB Title compound I, a fluorescent labeling agent, was synthesized from 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline, via chlorization the di-Me group, hydrolyzation to the ester, forming the disodium salt, after acidifying, giving the corresponding dicarboxylic acid, further sulfonation, got the product with high yield. The structure and purity of product was characterized by the m.p., IR, 1H NMR, UV spectrum, element anal..

RX(15) OF 15 - REACTION DIAGRAM NOT AVAILABLE

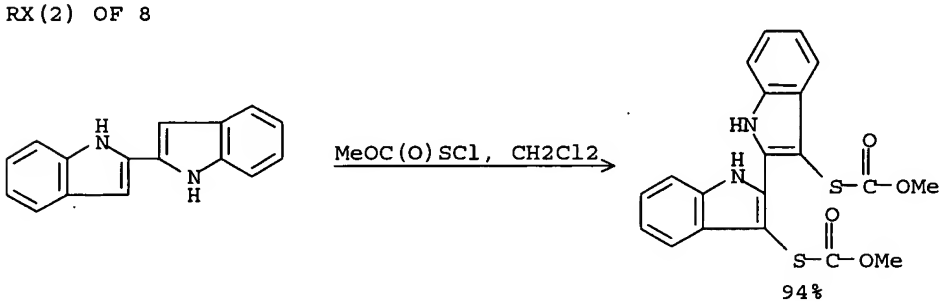
L77 ANSWER 2 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN 137:201292 CASREACT
TI Synthetic, spectroscopic, and X-ray crystallographic studies of [1,2,7,8]tetrathiacyclododecino[4,3-b:5,6-b':10,9-b'':11,12-b''']tetraindoles
AU Janosik, Tomasz; Bergman, Jan; Romero, Ivan; Stensland, Birgitta; Stalhandske, Claes; Marques, M. Manuel B.; Santos, Maria M. M.; Lobo, Ana M.; Prabhakar, Sundaresan; Duarte, M. Filomena; Florencio, M. Helena
CS Unit for Organic Chemistry, CNT, Department of Biosciences at Novum, Karolinska Institute, Huddinge, 141 57, Swed.
SO European Journal of Organic Chemistry (2002), (8), 1392-1396
CODEN: EJOCFK; ISSN: 1434-193X

PB Wiley-VCH Verlag GmbH
DT Journal
LA English
GI

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB Two conformationally different [1,2,7,8]tetrathiacyclododecino[4,3-b:5,6-b':10,9-b'': 11,12-b''']tetraindoles (I) and (II) were isolated in good yields, and the existence of a third conformer in solution was demonstrated by mass spectrometry and 1H NMR spectroscopy. The interconversions of the tetraindoles was studied. The conformation of II was confirmed by x-ray crystallog., while the conformations of I and II were assigned on the basis of spectroscopic data, and were also supported by mol. modeling studies. In addition, the elusive [1,2]dithiino[4,3-b:5,6-b']diindole-N,N-dimethylacetamide was isolated and the structure was proven by x-ray crystallog.

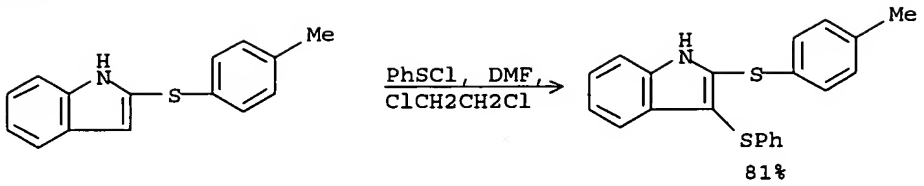
RX(2) OF 8



RE.CNT 22 THERE ARE 22 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

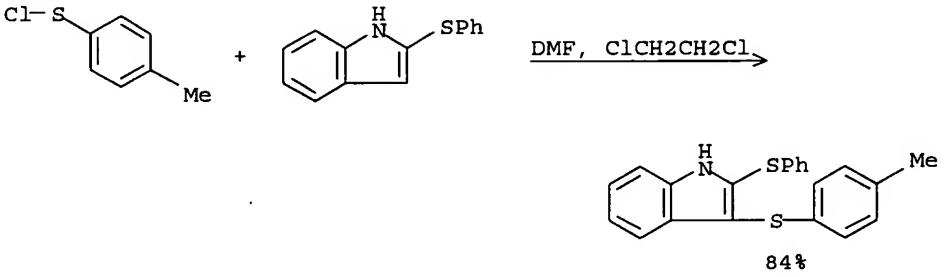
L77 ANSWER 3 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN 136:401369 CASREACT
TI Mechanism of the Second Sulfenylation of Indole
AU Hamel, Pierre
CS The Merck Frosst Centre for Therapeutic Research, Pointe-Claire-Dorval, QC, H9R 4P8, Can.
SO Journal of Organic Chemistry (2002), 67(9), 2854-2858
CODEN: JOCEAH; ISSN: 0022-3263
PB American Chemical Society
DT Journal
LA English
AB Sulfenylation of indole using a sulfenyl chloride occurs initially at the 3-position of the ring, leading to a 3-indolyl sulfide. When an excess of sulfenyl chloride is used, a second sulfide group is introduced at the 2-position, and an indolyl 2,3-bis-sulfide results. We have demonstrated that this second sulfenylation occurs not by direct introduction of the second sulfide at the 2-position but via initial formation of an indolenium 3,3-bis-sulfide intermediate, followed by migration of one of the sulfide groups to the 2-position. This was achieved by the isolation of two examples of 3H-indole 3,3-bis-sulfides and by subsequent demonstration that they rearrange to the indolyl 2,3-bis-sulfides by treatment with sulfenyl halides.

RX(3) OF 11



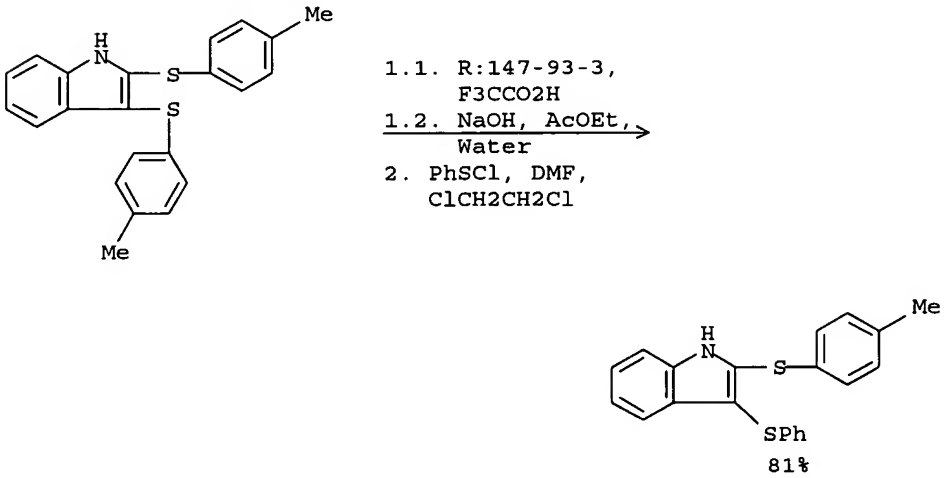
NOTE: in-situ generated reactant

RX(4) OF 11



NOTE: in-situ generated reactant

RX(10) OF 11 - 2 STEPS



NOTE: 2) in-situ generated reactant

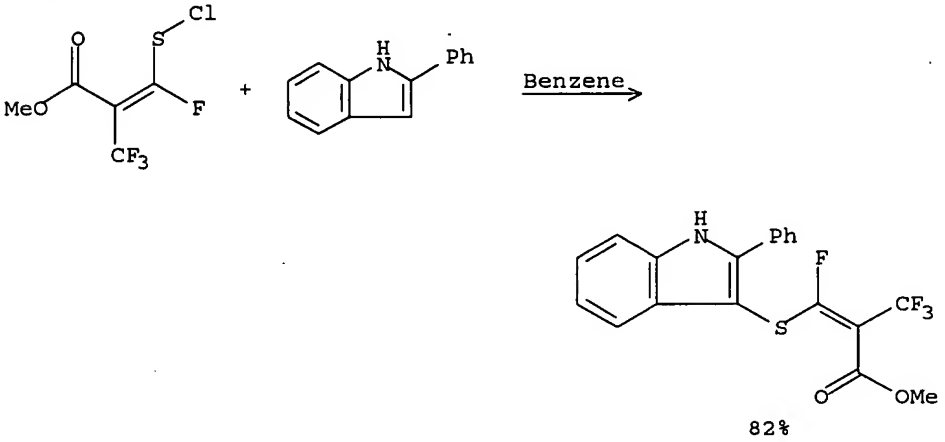
RE.CNT 14 THERE ARE 14 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L77 ANSWER 4 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN 136:369255 CASREACT
TI Reactions of 1,3,3,3-tetrafluoro-2-methoxycarbonylpropenylsulfenyl chloride with aromatic and heterocyclic compounds
AU Kovregin, A. N.; Sizov, A. Yu.; Ermolov, A. F.
CS Military University of Radiational, Chemical, Defence, Moscow, 107005, Russia
SO Russian Chemical Bulletin (Translation of Izvestiya Akademii Nauk, Seriya Khimicheskaya) (2001), 50(7), 1255-1258

Search done by Ross Schipe

CODEN: RCBUEY; ISSN: 1066-5285
PB Kluwer Academic/Consultants Bureau
DT Journal
LA English
AB 1,3,3,3-Tetrafluoro-2-methoxycarbonylpropenylsulfenyl chloride [i.e., 3-(chlorothio)-3-fluoro-2-(trifluoromethyl)-2-propenoic acid Me ester (I)] readily reacts with activated aromatic and heterocyclic compds. to form C-sulfenylation products as E isomers. In some cases, the reaction of I with phenolic compds. was accompanied by cyclization giving rise to fused 2-(2,2,2-trifluoro-1-methoxycarbonylethylidene)-1,3-oxathioles.

RX(4) OF 19

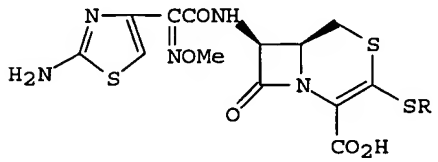


82%

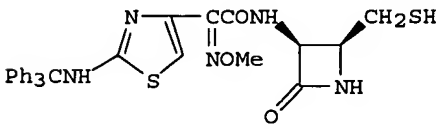
NOTE: stereoselective

RE.CNT 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L77 ANSWER 5 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN **112:76699** CASREACT
TI Synthesis of 3-(alkyl and aryl)thio-2-isocephems
AU Aszodi, Jozsef; Chantot, Jean Francois; Collard, Jeannine; Teutsch, Georges
CS Cent. Rech. Roussel Uclaf, Romainville, 93230, Fr.
SO Heterocycles (1989), 28(2), 1061-76
CODEN: HTCYAM; ISSN: 0385-5414
DT Journal
LA English
GI



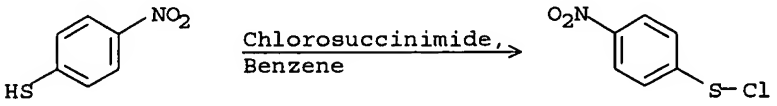
I



II

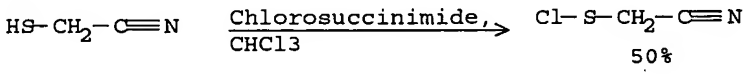
AB Isocephems I (R = Ph, 4-MeOC6H4, 4-O2NC6H4, 1-methyl-5-tetrazolyl, 5-methyl-1,3,4-thiadiazol-2-yl, CH2CO2H, CH2CH2NH2, CH2C6H4NO2-4) were prepared from the mercaptomethylazetidinone II and RSCHClCOCO2CMe3, prepared from RSU and N2CHCOCO2CMe3. Acid-catalyzed decarboxylation was a side-reaction in the deblocking step. Some I were more active against Staphylococci than cefotaxime, but I had poor activity against gram-neg. organisms.

RX(3) OF 85



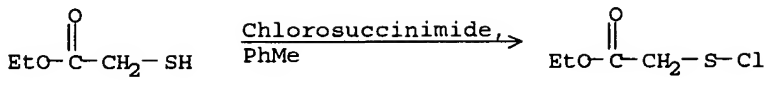
L77 ANSWER 6 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN 111:173585 CASREACT
TI Thioformyl cyanide. Gas-phase synthesis and millimeter wave spectrum
AU Bogey, Marcel; Demuynck, Claire; Destombes, Jean Luc; Gaumont, Annie; Denis, Jean Marc; Vallee, Yolande; Ripoll, Jean Louis
CS Lab. Spectrosc. Hertzienne, Univ. Lille I, Villeneuve d'Ascq, 59655, Fr.
SO Journal of the American Chemical Society (1989), 111(19), 7399-402
CODEN: JACSAT; ISSN: 0002-7863
DT Journal
LA English
AB The monomeric thioformyl cyanide HCSCN has been generated in the gas phase, either by flash-thermolytic retro-ene reaction of allylcyanomethyl sulfide or by vacuum gas-solid dehydrochlorination of cyanomethylsulfenyl chloride over potassium carbonate. An investigation by millimeter wave spectroscopy led to the unambiguous identification, mol. consts., and lifetime of HCSCN.

RX(5) OF 8

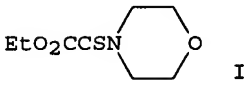


L77 ANSWER 7 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN 111:57025 CASREACT
TI Reactions of Wittig reagents with episulfides or elemental sulfur
AU Okuma, Kentaro; Tachibana, Yuji; Sakata, Junichi; Komiya, Takashi; Kaneko, Isao; Komiya, Yasuo; Yamasaki, Yusuke; Yamamoto, Shinichi; Ohta, Hiroshi
CS Fac. Sci., Fukuoka Univ., Fukuoka, 814-01, Japan
SO Bulletin of the Chemical Society of Japan (1988), 61(12), 4323-7
CODEN: BCSJA8; ISSN: 0009-2673
DT Journal
LA English
AB The reactions of Wittig reagents with episulfides gave sym. olefins and Ph3PS in moderate yields. The same olefins were obtained by reactions of Wittig reagents with elemental S. These reactions proceed through thiocarbonyl intermediates, the existence of which was confirmed by Diels-Alder reactions with dienes.

RX(3) OF 20



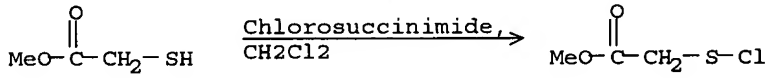
L77 ANSWER 8 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN 109:230907 CASREACT
TI Preparation of carboxythioamides by the reaction of thioaldehydes with secondary amines
AU Okuma, Kentaro; Honda, Takumi; Ohta, Hiroshi
CS Fac. Sci., Fukuoka Univ., Fukuoka, 814-01, Japan
SO Fukuoka Daigaku Rigaku Shuho (1987), 17(2), 41-4
CODEN: FDRSDG; ISSN: 0386-118X
DT Journal
LA English
GI



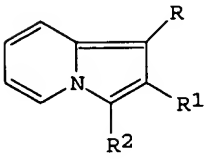
I

AB The reactions of thioaldehydes with amines were carried out. When carboxymethanesulfenyl chlorides were treated with secondary amines in the presence of Et3N, carboxythioamides, e.g., I, were obtained. Ordinary sulfenylchlorides reacted with secondary amines to give corresponding sulfenamides.

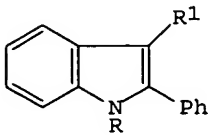
RX(12) OF 14



L77 ANSWER 9 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN 106:49947 CASREACT
TI Electron-transfer processes: sulfenylation of electron-rich systems by diaryl disulfides and aryl thiosulfonates
AU Berti, Corrado; Colonna, Martino; Poloni, Marino
CS Fac. Ing., Univ. Bologna, Bologna, I-40136, Italy
SO Gazzetta Chimica Italiana (1986), 116(4), 181-4
CODEN: GCITA9; ISSN: 0016-5603
DT Journal
LA English
GI



I

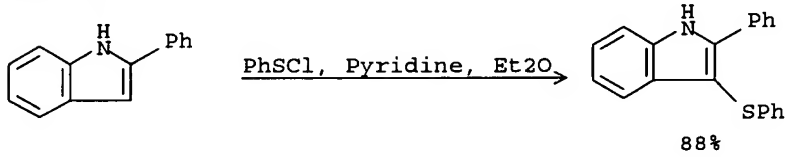


IV

AB Sulfenylation of indolizine I (R = Me, R1 = Ph, R2 = H) with PhSSPh (II) in refluxing EtOH gave I (R2 = SPh). Dibenzothiazol-2-yl disulfide (III),

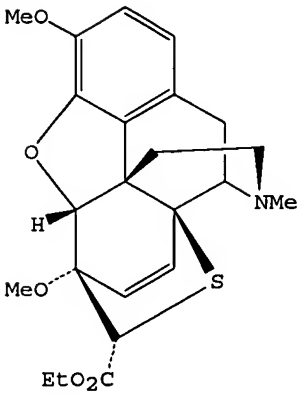
a compound with a low S-S bond energy, reacted with I (R = Me, R1 = Ph, R2 = H; R = H, R1 = Ph, R2 = Me) at room temperature giving I (R = Me, R1 = Ph, R2 = benzothiazol-2-ylthio; R = benzothiazol-2-ylthio, R1 = Ph, R2 = Me) in 66-92% yields. Indoles IV (R = H, Me; R1 = H), which have a higher oxidation potential than indolizines, reacted with II only on irradiation The sulfenylation of IV can be easily obtained either by activation with AgNO3 or using III. PhSSO2C6H4Cl-4 sulfenylated I (R = Me, R1 = Ph, R2 = H) and IV at room temperature All these reactions are discussed in terms of electron-transfer processes.

RX(10) OF 15

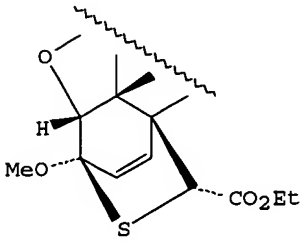


88%

L77 ANSWER 10 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN 104:5547 CASREACT
TI Ethyl and methyl thioxoacetates, dienophilic thioaldehydes formed from sulfenyl chlorides by 1,2-elimination
AU Bladon, Christine M.; Ferguson, Irene E. G.; Kirby, Gordon W.; Lochead, Alistair W.; McDougall, Duncan C.
CS Dep. Chem., Univ. Glasgow, Glasgow, G12 8QQ, UK
SO Journal of the Chemical Society, Perkin Transactions 1: Organic and Bio-Organic Chemistry (1972-1999) (1985), (7), 1541-5
CODEN: JCPRB4; ISSN: 0300-922X
DT Journal
LA English
GI



III



IV

AB Treatment of RO2CCH2SCl (I; R = Me, Et) with Et3N at room temperature gave the corresponding RO2CCHS (II). Generation of transient II (R = Et) in the presence of conjugated dienes gave the corresponding cycloadducts. E.g., treatment of I (R = Et) with Et3N in C6H6-MeOH containing thebaine at room temperature gave 67% cycloadduct III, which isomerized at 111° to the more stable adduct IV by dissociation and recombination. Cycloadducts of II (R = Et) and anthracene or 9,10-dimethylanthracene similarly dissociated at 111°, providing a clean and convenient source of II (R = Et).

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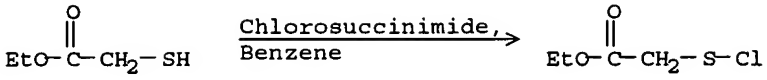
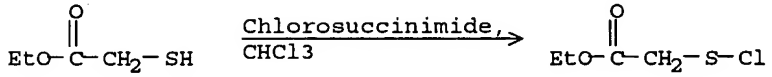
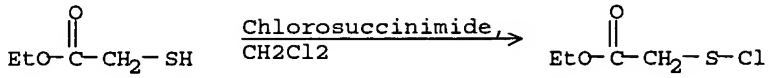
RX(1) OF 58



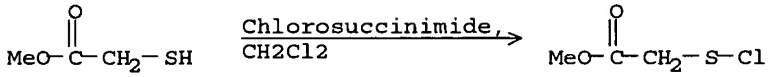
RX(2) OF 58



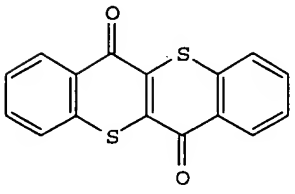
RX(3) OF 58



RX(4) OF 58



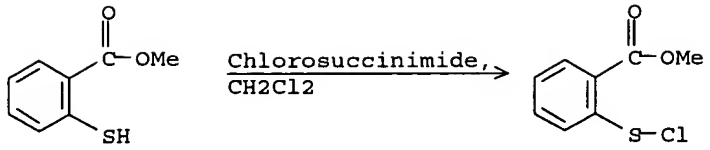
L77 ANSWER 11 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN 103:123385 CASREACT
TI Thipyranothiopyran chemistry. 5. Synthesis of dibenzo[b,g]thiopyrano[3,2-b]thiopyran-6,12-dione (thioepindolidione)
AU Chen, Chin H.; Fox, John L.
CS Res. Lab., Eastman Kodak Co., Rochester, NY, 14650, USA
SO Journal of Organic Chemistry (1985), 50(19), 3592-5
CODEN: JOCEAH; ISSN: 0022-3263
DT Journal
LA English
GI



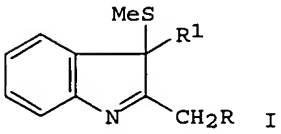
I

AB Thioepindolidione (I), a derivative of a thiopyrano[3,2-b]thiopyran, was synthesized along with trans-thioindigo from thiochroman-4-one. The absorption and fluorescence characteristics and chromogenic properties of I are compared with those of thioindigo and epindolidione.

RX(2) OF 28



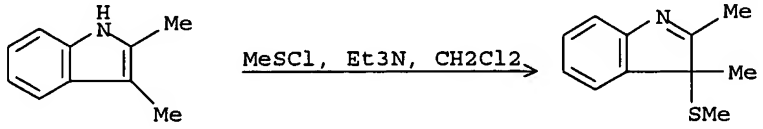
L77 ANSWER 12 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN 103:37318 CASREACT
TI Methanesulfenylation of 2,3-dialkylindoles: synthesis and reactions of 3-methylthioindolenines
AU Friesen, Richard W.; Vice, Susan F.; Findlay, C. Edward; Dmitrienko, Gary I.
CS Guelph-Waterloo Cent. Grad. Work Chem., Univ. Waterloo, Waterloo, ON, N2L 3G1, Can.
SO Tetrahedron Letters (1985), 26(2), 161-4
CODEN: TELEAY; ISSN: 0040-4039
DT Journal
LA English
GI



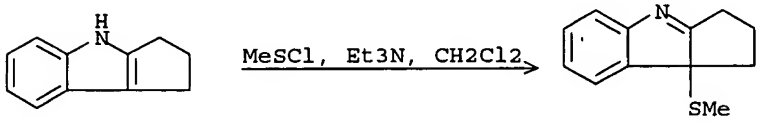
I

AB 3-Methylthioindolenines I [R = H, R1 = Me, RR1 = (CH2)2, (CH2)3], readily prepared from 2,3-dialkylindoles and MeSCl, are heat and acid labile, yielding the parent indole and MeSSMe as the major decomposition products, and are readily reduced to the parent indoles with mercaptoacetic acid.

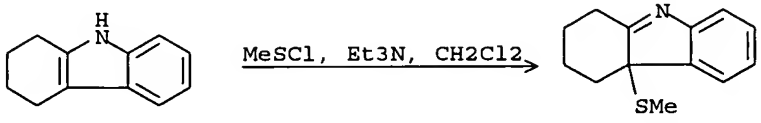
RX(8) OF 12



RX(9) OF 12

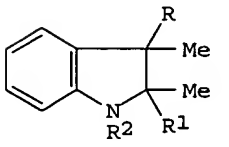


RX(10) OF 12



Search done by Ross Schipe

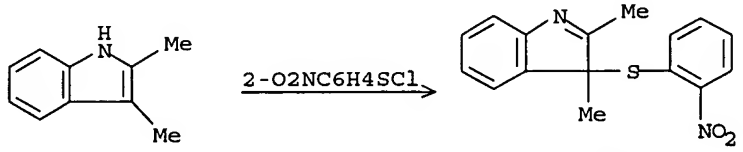
L77 ANSWER 13 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN 97:23571 CASREACT
TI Arylsulfenylation of 2,3-dialkylindoles. Preparation of 3-arylthioindolenines and thermal conversion to an N-sulfenylindole
AU Dmitrienko, Gary I.; Friesen, Richard W.; Carson, Loraine; Vice, Susan F.
CS Guelph-Waterloo Cent. Grad. Work Chem., Univ. Waterloo, Waterloo, N2L 3G1, Can.
SO Tetrahedron Letters (1982), 23(8), 821-4
CODEN: TELEAY; ISSN: 0040-4039
DT Journal
LA English
GI



II

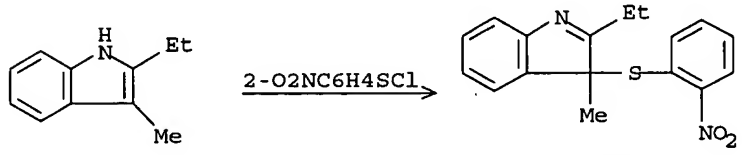
AB Reaction of ClSC6H4NO2-o (I) with 2,3-dialkylindoles gave 3-(o-nitrophenylsulfenyl)-2,3-dialkylindolenines which underwent thermal rearrangement to give N-(o-nitrophenylsulfenyl)indoles. Thus, I reacted with 2,3-dimethylindole in the presence of Et3N to give 90% indole II (R = SC6H4NO2-o, R1R2 = bond) (III). Heating III at 95° for 15 min gave 14% II (RR1 = bond, R2 = SC6H4NO2-o).

RX(2) OF 10



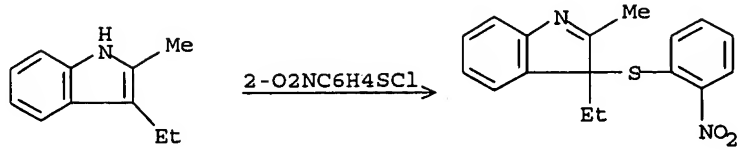
90%

RX(3) OF 10



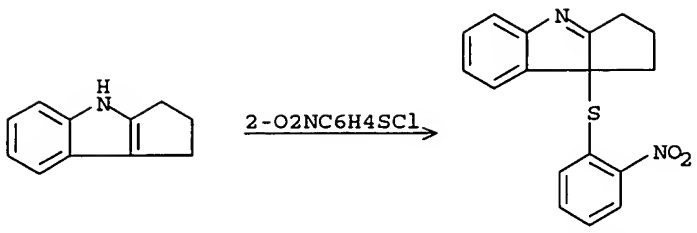
85%

RX(4) OF 10



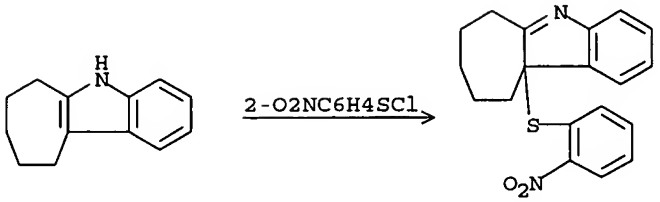
75%

RX(5) OF 10



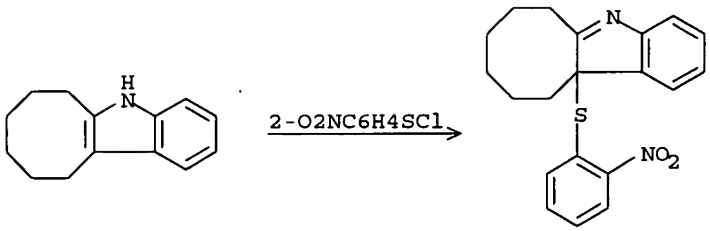
92%

RX(6) OF 10



92%

RX(7) OF 10



98%

L77 ANSWER 14 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN 95:187068 CASREACT
TI Antidiabetic pyrrolecarboxylic acids
IN Holland, Gerald F.
PA Pfizer Inc., USA
SO U.S., 28 pp.
CODEN: USXXAM
DT Patent
LA English
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 4282242	A	19810804	US 1980-128119	19800307
	US 4351843	A	19820928	US 1981-256933	19810423
	US 4511575	A	19850416	US 1982-406306	19820809
	US 4511576	A	19850416	US 1982-406921	19820810
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	US 1981-256933	19810423			

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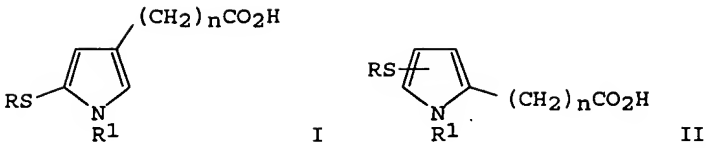
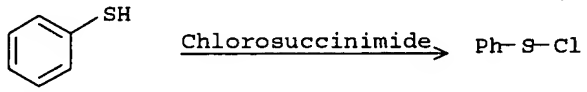


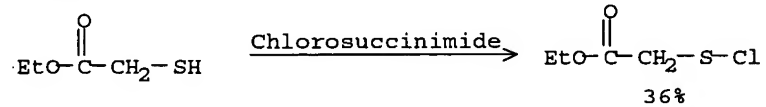
AB Pyrrolecarboxylates I and II (R = alkyl, CH2Ph, optionally substituted Ph; R1 = H, Me, Et; n = 0, 1) were prepared Thus CH2:CHCO2Me was treated with 4-MeC6H4SO2CH2NC to give Me 3-pyrrolecarboxylate which was treated with PhSCl and saponified to give I (R = Ph, R1 = H, n = 0).

RX(1) OF 39



L77 ANSWER 15 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN 93:7609 CASREACT
TI Synthesis of aliphatic sulfenyl halides containing ester groups
AU Seliger, Hartmut; Goertz, Hans Helmut
CS Sekt. Polym., Univ. Ulm, Ulm, D-7900, Fed. Rep. Ger.
SO Synthetic Communications (1980), 10(3), 175-82
CODEN: SYNCAV; ISSN: 0039-7911
DT Journal
LA English
AB Aliphatic sulfenyl halides RCO2CH2CH2SR1 (I, R = H, Me, Et, F3C; R1 = Cl, Br), AcOCHR2CHR3SR1[II, R1 = Cl, Br; R2 = Me, Et, R3 = H; R2R3 = (CH2)4], and EtO2C(CH2)n CHR4SR1 (III, R1 = Cl, Br; n = O, R4 = H, Me; n = 1, R4 = CO2Et) were prepared I-III (R1 = H) were treated with N-chlorosuccinimide to give 31% to 72% I-III (R1 = Cl). I-III (R1 = Br) were prepared similarly, but decomposed on heating to the corresponding disulfides with elimination of Br.

RX(14) OF 67



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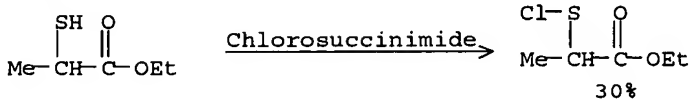

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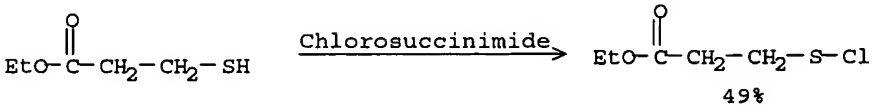

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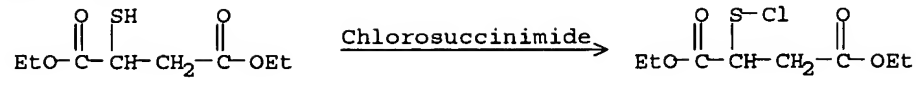

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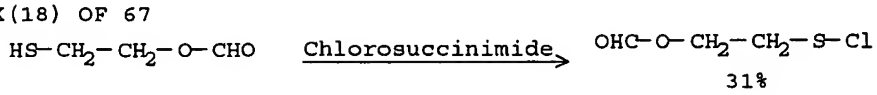

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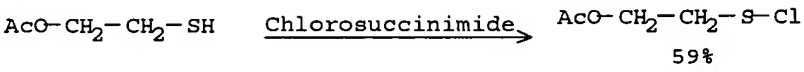

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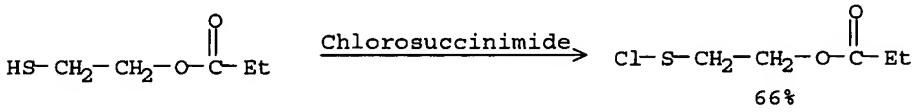

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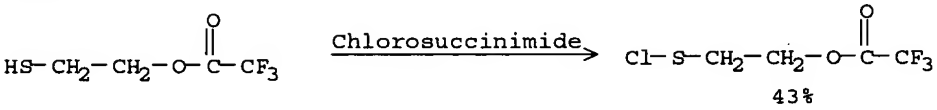

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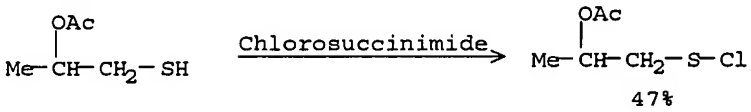

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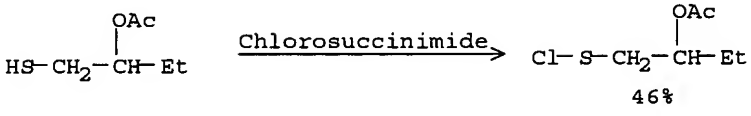

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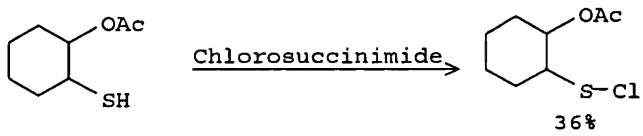

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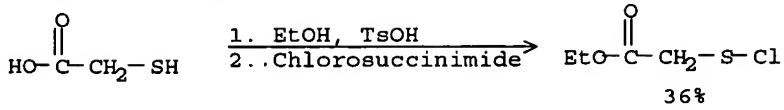

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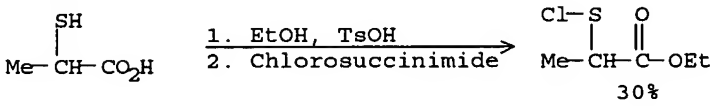

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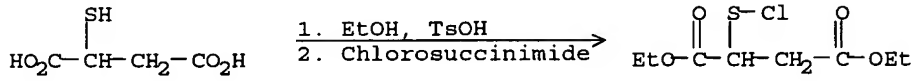

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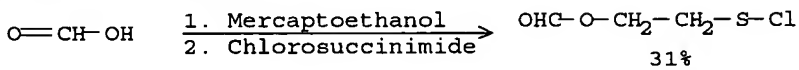

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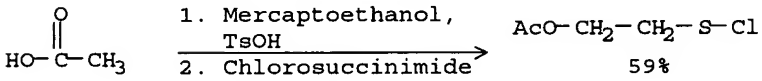

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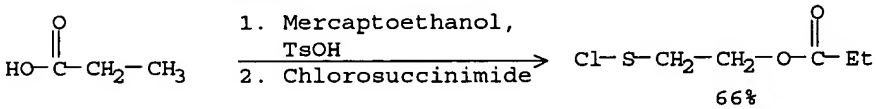

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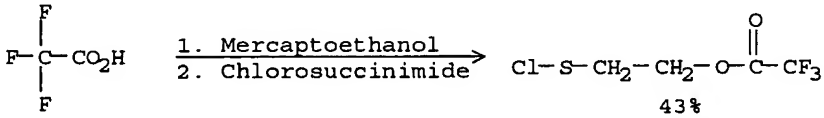

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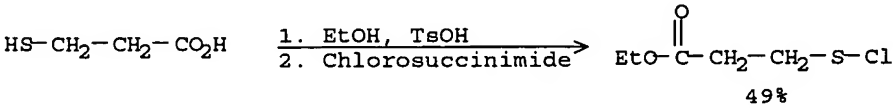

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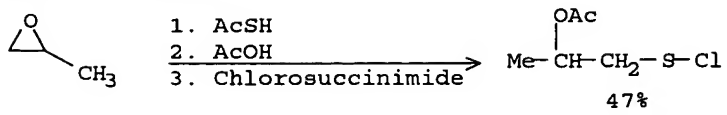
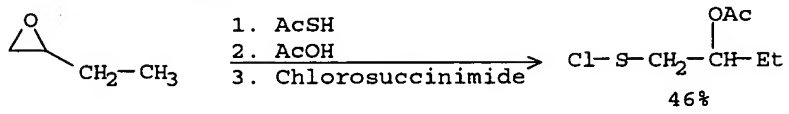


RX(64) OF 67 - 3 STEPS



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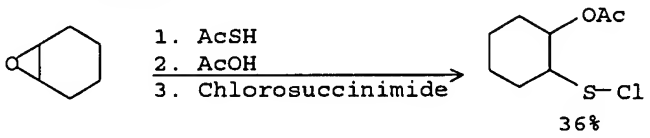
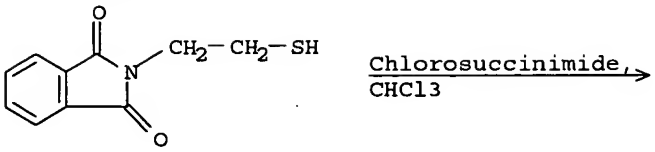
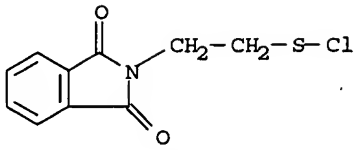


L77 ANSWER 16 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN 67:63971 CASREACT
TI A search for agents protecting against irradiation IX. Synthesis of some asymmetric disulfides
AU Tulecki, Jerzy; Dabrowski, Jerzy; Kalinowska-Torz, Jadwiga
CS Katedra Tech. Chem. Srodkow Leczniczch, Akad. Med., Poznan, Pol.
SO Dissertationes Pharmaceuticae et Pharmacologicae (1966), 18(5), 473-8
CODEN: DPHFAK; ISSN: 0012-3870
DT Journal
LA Polish
GI For diagram(s), see printed CA Issue.
AB Bromoethylphthalimide (74.2 g.) and 22.3 g. thiourea in 240 ml. 90% EtOH was refluxed 4 hrs., the dry residue dissolved in 300 ml. H2O and treated with 0.5 g. Na2SO3 and then with 16.5 g. KOH in 100 ml. H2O, and the mixture heated 30 min. on a steam bath to give 50.4 g. 2-mercaptoethylphthalimide (I), m. 74-5.5°; 2,4-dinitrophenyl derivative m. 186-7°. I (2.2 g.) in 70 ml. CHCl3 treated with 1.4 g. N-chlorosuccinimide (26% Cl+) gave, after 10 min., phthalimide-N-ethylsulfenyl chloride (II), which when mixed with 1.6 g. thiosalicylic acid in 130 ml. Et2O gave 77% o-carboxyphenyl 2-phthalimidoethyl disulfide (III), m. 208-12° and 219-21°. II, obtained from 4.8 g. I, in 70 ml. CHCl3 was kept with 3.8 g. 2-mercaptothiazole in 200 ml. CHCl3 10 min. and refluxed 10 min. to give 69.5% 2-benzothiazolyl 2-phthalimidoethyl disulfide (IV), m. 133-4°. Similarly, 68.3% 2-furfuryl 2-phthalimidoethyl disulfide, m. 95-5.5°, was obtained from II and furfuryl mercaptan. Thiosalicylic acid (2.7 g.) in 30 ml. CHCl3 added dropwise to 2.5 g. N-chlorosuccinimide in 60 ml. CHCl3 gave the sulfenyl chloride, which treated with 3-phenyl-propyl mercaptan in 30 ml. CHCl3 at room temperature and kept 20 min. yielded 62% o-carboxyphenyl 3-phenylpropyl disulfide (V), m. 100-100.5°. Cf. CA 67: 53825n.

RX(2) OF 2



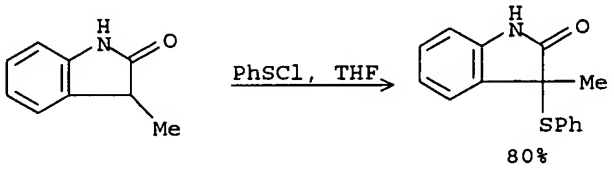


NOTE: Classification: Chlorination; # Conditions: NCS CHCl3; 10mn; # Comments: also C.A., 67, 6002 (1967)

L77 ANSWER 17 OF 17 CASREACT COPYRIGHT 2005 ACS on STN
AN 63:38959 CASREACT
TI Indoles for comparison with amanita poisons. V. Thio ether syntheses in the oxindole, indoline, and indole series
AU Wieland, Theodor; Grimm, Dieter
CS Univ. Frankfurt, Germany
SO Chemische Berichte (1965), 98(6), 1727-35
CODEN: CHBEAM; ISSN: 0009-2940
DT Journal
LA German
AB cf. CA 58, 11310c. The thioether grouping was introduced into 3-substituted oxindoles via the 3-Br derivative with mercaptides or by direct substitution of H with sulfenyl chlorides. The resulting oxindole 3-thio ethers could not be reduced to the corresponding indolines since the Scontg. substituent was cleaved off by the LiAlH4. The introduction of thio ether groups into the 2-position of 3-substituted 6-methoxyindoles was achieved with sulfenyl chlorides; the resulting products showed uv spectra very similar to that of amanitine. 3-Methyloxindole (I) (7.3 g.) in 200 cc. CCl4 treated dropwise with 8 g. Br in 400 cc. CCl4 yielded 10 g. 3-Br derivative (II) of I, m. 220-2° (decomposition). II (5.65 g.) in 40 cc. dry tetrahydrofuran refluxed 2 hrs. with 2.10 g. EtSNa and kept overnight gave 4.0 g. 3-EtS derivative (III) of I, m. 74° (repptd. from EtOH with H2O). III (2.07g.) in 30 cc. dry dioxane heated 3 hrs. with 0.37 g. LiAlH4 in 10 cc. dry Et2O gave skatole, m. 86-8° (aqueous MeOH). PhSH (d. 1.078)(1.1 g.) added with cooling to 1.34 g. N-chlorosuccinimide in 2 cc. dry CHCl3 and 3 cc. dry tetrahydrofuran gave a solution of PhSCl (IV). The PhSCl solution and 1.47 g. I in tetrahydrofuran kept 1 hr. at room temperature yielded 2.1 g. 3-PhS derivative of I, m. 163° (aqueous MeOH). IV from 1.02 g. PhSH and 1.35 g. N-chlorosuccinimide treated 2 hrs. at room temperature with 0.7 g. 1-methyloxindole (V) gave 1.0 g. 3-PhS derivative of V, m. 118° (aqueous MeOH). 5-Ethoxy-1,3-dimethyloxindole (VI) (2.0 g.) in 10 cc. tetrahydrofuran treated 2 hrs. with IV from 1.02 g. PhSH and 1.35 g. N-chlorosuccinimide gave 2.0 g. 3-PhS derivative of VI, m. 60° (aqueous MeOH). Trimeric 3,3-dimethylindolenine (4.34 g.) in 60 cc. C6H6 and 3.44 g. BzCl treated 2 hrs. at room temperature with 0.69 g. Na in 15 cc. EtSH yielded 7.0 g. 2-ethylthio-3,3-dimethyl-1-benzoylindoline, b0.01 165-8°. AcEtCHCO2Et (40 g.) dissolved at -15° in 190 cc. 20% aqueous NaOH and 190 cc. EtOH, treated at -10° with diazotized 30.9 g. m-MeOC6H4NH2, and stirred 0.5 hr. at -4° and a 60-g. portion of the resulting 65 g. oily hydrazone refluxed 45 min. with 300 cc. absolute EtOH and 45 cc. concentrated H2SO4 yielded 21 g. 2-carbethoxy-3-methyl-6-methoxyindole (VII), m. 122° (petr. ether). VII (15 g.), 187 cc. 10% aqueous NaOH, and 50 cc. EtOH refluxed 5 hrs. yielded 14 g. 2-CO2H analog (VIII) of VII, m. 202° (aqueous EtOH). VIII heated 0.5 hr. at

220° gave about 70% 3-methyl-6-methoxyindole (IX), m. 127° (aqueous MeOH). IX (1.92 g.) in 20 cc. dry Et2O treated dropwise with stirring under N at 0° with 1.2 g. EtSCl in 20 cc. CHCl3 and kept 2 hrs. at 0° and 2 hrs. at room temperature yielded 1.42 g. X (R = Me, R' = Et) (XI), m. 138° (sublimed at 130-40°/0.005 mm.). The S-Cl derivative from 2.55 g. N-carbobenzyloxycysteine (XII) and 1.35 g. N-chlorosuccinimide (XIII) treated with 1.61 g. IX yielded 2.5 g. X [R = Me, R' = PhCH2O2CNHCH(CO2H)CH2], decomposed from 90° (Et2O-petr. ether). Diethyl acetamido(6-methoxy-3-indolylmethyl)malonate (0.752 g.), m. 145°, treated with the S-Cl derivative from 0.51 g. XII and 0.27 g. XIII in 6 cc. dry CHCl3 and 4 cc. dry tetrahydrofuran yielded 1.0 g. X [R = AcNHC(CO2Et)2CH2, R' = PhCH2O2CNHCH(CO2H)CH2] (XIV), m. 103° (Et2O-petr. ether). The uv spectra of IX, XI, VIII, and XIV are recorded.

RX(1) OF 2



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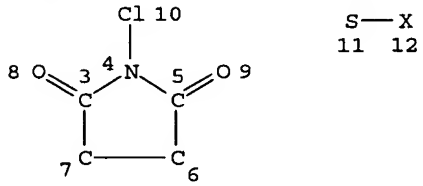

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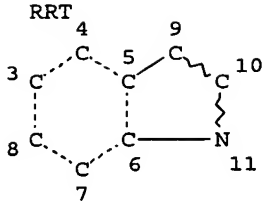
RRT

RRT



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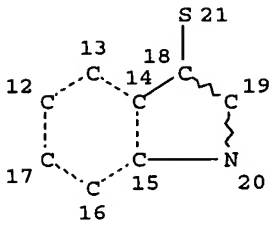

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NUMBER OF NODES IS 21

STEREO ATTRIBUTES: NONE
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